# EVAPORATION OF SEWAGE PLANT EFFLUENT

By
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A DISSERTATION PRESENTED TO THE GRADUATE COUNCIL OF
THE UNIVERSITY OF FLORIDA
IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE
DEGREE OF DOCTOR OF PHILOSOPHY

UNIVERSITY OF FLORIDA 1970



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### DEDICATION

This dissertation is dedicated to the memory of Dr. Charles I.
Harding. I first met Professor Harding in 1967, shortly after I began
my graduate study at the University of Florida. I was impressed with
the way he frequently presented to his classes specific problems
encountered by the practicing engineer and tried to prepare us to
effectively deal with them. When he left the University to enter private
practice, his interest in students and education continued. It was
Dr. Harding who approached us at the University and proposed that we
consider a research project on the evaporation of sewage treatment plant
effluent. When we responded favorably, he further assisted by committing
his firm to provide the initial financing to begin construction of the
needed research equipment. This was long before there were any
assurances of government research funds to help finance the project.
His enthusiastic help and leadership were absolutely essential to this
project.

I last saw Dr. Harding in a conference for consulting engineers and university professors. His commitment to education was again evident as he unselfishly shared his knowledge of the world of consulting engineering.

Dr. Harding was a true engineer. To him, problems existed only to be solved, not just theoretically solved, but eliminated by the full scale application of the theory. I am proud to have had the privilege of working with him and to dedicate this work to his memory.

#### ACKNOWLEDGEMENTS

I would like to acknowledge first, my committee chairman,
Dr. J. E. Singley. Throughout my entire graduate program he has
been not only my principal teacher and advisor, but also a personal
friend. His encouragement and support have been invaluable.

As I expressed in the dedication, this work would not even have been started had it not been for Dr. C. I. Harding.

Thanks go also to the other members of my committee, Professor

T. deS. Furman and Dr. J. D. Winefordner, for their help and support.

Throughout this project, much has depended on our being able to promptly secure goods and services both from within and outside of the University. With his complete knowledge of administrative procedures and willingness to help, Dr. W. H. Morgan has been an invaluable enabler.

I wish to thank Mr. Hugh Holburn and Mr. Stanley Ridgeway of Indian River Construction Company for all their help in the design and construction of the evaporator. They are responsible for the final construction of the unit and they did an excellent job.

I express my thanks to Dr. R. S. Sholtes and Mr. Howard McGraw for their help in setting up the evaporator instrumentation.

The volume of analytical work in this project was staggering, and I wish to thank the three chemists who did such a fine job, Mr. Ryosuke Miura, Mr. Roger Yorton, and Mr. Gary Ashley.

For their many long hours of help in operating the evaporator,  ${f I}$  wish to thank Mr. Donald Winsor and Mr. Gary Ashley.

My thanks also to Mrs. Jeanne Dorsey for typing this final manuscript.

I wish to acknowledge the financial support of the Department of the Interior through Federal Water Quality Administration Grant No. 5T1-WP-119-03 and Contract No. 14-12-571.

Last, but by no means least, I wish to express my thanks to my wife, Rubye, and my son, Jim. Their help and contributions to this whole effort cannot be measured nor can my thanks to them be expressed in words.

# TABLE OF CONTENTS

		Page
ACKNOWLE	DGEMENTS	iii
LIST OF	TABLES	vii
		AT1
LIST OF	FIGURES	ix
ABSTRACT		х
CHAPTER		
I.	INTRODUCTION	1
	General Background	,
	Previous Work on Evaporation of Treated Sewage	1 5
	Objectives of this Research	9
		,
II.	THEORETICAL BACKGROUND	10
	Separation by Evaporation	10
	Volatile Organics	11
	Ammonia	12
	Scaling and Fouling	17
	Calcium Carbonate	18
	Calcium Sulfate	20
	Magnesium Hydroxide	20
	Silica	22
	Organic Scale	23
	Bacteria or Virus Contamination	23
	Ultimate Disposal	25
III.	RESEARCH PLAN	27
	Experimental Equipment	27
	Effluents Available	28
	Experimental Plan	29
	Analytical Plan and Procedures	30
IV.	DESCRIPTION OF EQUIPMENT	35
	Degasser	35
	Evaporator	38
	Carbon Column	45
	Ammonia Column	45

		Page
V.	EXPERIMENTAL RESULTS	46
	Initial Testing	46
	Extended Aeration Plant Effluent	49
	Contact Stabilization Plant Effluent	53
	Trickling Filter Plant Effluent	53
	Activated Carbon Tests	60
	Ammonia Ion Exchange Tests	61
	Aeration Tests	62
	Product Water Conductivity	62
	Scaling During Check-Out and Initial Testing	64
	Long Term Runs	66
	First Extended Aeration Effluent Run	67
	Trickling Filter Effluent Run	71
	Three Effect Ammonia Distribution Tests	76 79
	Three Brices Amanonia Distribution lests	79
VI.	DISCUSSION OF RESULTS	83
	Product Quality	83
	Ammonia Content	83
	Chemical Oxygen Demand	85
	Odor	85
	Conductivity	86
	Nitrate	86
	Bacteriological Tests	86
	Evaporator Scaling	87
	Scaling by Extended Aeration Effluent	87
	Scaling by Trickling Filter Effluent	89
	Heat Transfer Coefficients	89
VII.	CONCLUSIONS	91
APPENDICE	S	93
A	anden v	
АРР	endix I	94
Ann	Test Results - Campus Sewage Treatment Plant	94
whh	endix IICalculation of Heat Transfer Coefficients	96
	Carcaración di neat itansfer Coefficients	96
LIST OF R	EFERENCES	99
BIOGRAPHI	CAL SKETCH	102

# LIST OF TABLES

T	ables	Page
1	. Typical Analyses of Sea Water and Secondary Effluent	2
2	. Disassociation Constants for Aqueous Ammonia and Water	14
. 3	. Analytical Tests on Liquid Samples	31
4	. Analytical Tests on Scale	34
5 : :	Evaporator Operating Conditions for Initial Single Stage	47
6	. Initial Tests, Extended Aeration Effluent, Vacuum Conditions	50
7	Initial Tests, Extended Aeration Effluent, Atmospheric Conditions	51
8.	Initial Tests, Extended Aeration Effluent, Pressure	52
9.	Initial Tests, Contact Stabilization Effluent, Vacuum Conditions	54
10.	Initial Tests, Contact Stabilization Effluent, Atmospheric Conditions	55
11.	Initial Tests, Contact Stabilization Effluent, Pressure Conditions	56
12.	Initial Tests, Trickling Filter Effluent, Vacuum Conditions	57
13.	Initial Tests, Trickling Filter Effluent, Atmospheric Conditions	58
14.	Initial Tests, Trickling Filter Effluent, Pressure Conditions	59
15.	Typical Analysis of Tap Water, Gainesville, Florida	65
16.	Analytical Results - First Extended Aeration Effluent Run	70
17.	Scale from First Extended Aeration Effluent Pun	70

Tabl	les	Page
18.	Analytical Results - Trickling Filter Effluent Run	73
19.	Scale from Trickling Filter Effluent Run	75
20.	Activated Carbon Treatment of Products from Trickling Filter Effluent Run	76
21.	Analytical Results - Second Extended Aeration Effluent Run	78
22.	Bacteriological Tests - Second Extended Aeration Effluent Run	80
23.	Scale from Second Extended Aeration Effluent Run	81
24.	Analytical Results - Three Effect Ammonia Distribution Tests	82
25.	Campus Sewage Treatment Plant Test Results, High Rate Trickling Filter	94
26.	Campus Sewage Treatment Plant Test Results, Contact Stabilization Plant	95

# LIST OF FIGURES

	Figure		Page
	1.	Distribution of NH $_3$ and NH $_4^+$ as a Function of pH and Temperature	15
	2.	Calcium Sulfate Solubility	21
	3.	Overall Flowsheet	36
	4.	Degassing System Flowsheet	37
	5.	Evaporator Flowsheet	39
	6.	Evaporator	40
	7.	Vapor-Liquid Separator	42
	8.	Product Receiver System Flowsheet	44
	9.	Conductivity vs. Ammonia Concentration	63
1	0.	Product Rates, First Extended Aeration Effluent Run	69
1	1.	Overall Heat Transfer Coefficients	74

Abstract of Dissertation Presented to the Graduate Council of the University of Florida in Partial Fulfillment of the Requirements for the Degree of Doctor of Philosophy

# EVAPORATION OF SEWAGE PLANT EFFLUENT

bу

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August, 1970

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The technical feasibility of evaporation of sewage treatment plant effluent for the purpose of reuse was investigated. The process used was similar to one of the major processes used in sea water conversion, evaporation in a long tube vertical (LTV) evaporator. As evaporator feedwater, treated sewage has the advantage over sea water of having much lower total dissolved solids. This should result in higher evaporator operating temperatures, reduced scaling problems, and negligible boiling point rise. The major disadvantages of treated sewage as an evaporator feedwater are that the water usually contains volatile organic materials and ammonia that may contaminate the product.

The objectives of the research were (1) to define the relationships between product water quality, feedwater quality, and evaporation conditions, (2) to define the relationships between post evaporation polishing, feedwater quality, and evaporation conditions, and (3) to define the relationships between evaporator tube scaling, feedwater quality, and evaporation conditions. The experimental equipment consisted of a three effect LTV evaporator constructed of 316 stainless steel. Each effect was a single one inch diameter tube with a four inch diameter steam jacket. The effective heated length of the tube was 14 feet. Vapor-liquid separators atop each effect separated the vapor from the liquid. The evaporator could be operated as a single effect or triple effect unit. Possible operating conditions varied from around 28 inches Hg vacuum to about 55 psig. All evaporator feedwater was first degassed under vacuum to remove dissolved gases. Prior to degassing, the pH was adjusted to 4.9 so that all carbonates would be removed.

Feedwaters available to the evaporator included extended aeration effluent, high rate trickling filter effluent, and contact stabilization effluent. All three units treated raw waste from a large university complex.

The experimental work was divided into two principal phases. The first phase work was directed towards defining the product quality relationships with the various variables. The second phase work was directed towards defining the relationships between evaporator tube scaling and the various variables. Most of the first phase work was accomplished by making relatively short runs, three to five hours, using only one effect of the evaporator. Post treatment processes included aeration, activated carbon, and ammonia selective ion exchange. In the second phase, the evaporator was operated as a triple effect unit over an extended period of time. After each run, the evaporator tubes were mechanically cleaned and the scale analyzed.

Results of the first phase work showed that an odor-free product could not be produced from any of the three feedwaters under any

operating condition from 112°F to 290°F. The odor intensity increased as the evaporator operating temperature and/or the chemical oxygen demand of the feedwater increased. In all cases, post treatment with activated carbon removed all odors. Aeration would not remove all odors. Product produced under pressure conditions using trickling filter feed contained significantly more organic contamination than any other products. Product contamination by ammonia could not be controlled by adjusting the pH of the feed in the range 5.1 to 8.7. However, ammonia in the product water was removed by ion exchange.

The scaling evaluations were carried out under pressure conditions using extended aeration and trickling filter effluent. Trickling filter feedwater was judged unsuitable because of excessive scaling. Scaling problems with extended aeration feedwater were minor, and with post treatment by activated carbon, a high quality product water was produced.

Because of increased efficiencies due to higher operating temperatures and negligible boiling point rise, wastewater evaporation should be more economical than sea water evaporation.

#### I. INTRODUCTION

### General Background

The continuing increase in demand for potable water coupled with increasing amounts of pollutants being discharged into the environment has forced mankind to resort to poorer quality sources of water for a potable water supply. In some areas the availability of fresh water is so limited that we have turned to the sea for our water supply. Millions of dollars have been expended in research and development of saline water conversion processes. This research has resulted in 627 desalting plants worldwide which were either in operation or under construction as of 1968. The total capacity of these plants is 222 mgd, and distillation or evaporation accounts for 93% of this capacity. It is estimated that the desalting capacity may reach 1 bgd by 1975. It is now clearly evident that there are many places in the world where desalting is either the most economical or only source of potable water.

The research reported here examines the most common type of desalting process, distillation or evaporation, used with an alternate feedwater, sewage treatment plant effluent. This feedwater should be available in any area having a central sewage collection system and would in all cases be as close or closer to the consumers than sea water. Typical analyses of sea water and a secondary effluent are shown in Table 1.<sup>2,3</sup> The main differences between these two waters are

 ${\tt TABLE~1}$   ${\tt TYPICAL~ANALYSES~OF~SEA~WATER~AND~SECONDARY~EFFLUENT}$ 

	2	3	Increment 3
	Sea Water <sup>*</sup>	Secondary Effluent	Added
	ppm	mg/1	mg/1*
Chloride	19,353	130	75
Sodium	10,760	135	70
Magnesium	1,294	25	7
Sulfur	904	33	10
Calcium	413	60	15
Potassium	387	15	10
Gross Organics	_	55	52
BOD	-	25	25
Nitrate	0.04 - 3	15	10
Phosphate	0.003 - 0.3	25	25
Silica		50	15
Ammonia	-	20	20
Total Dissolved Solids	35,000	730	320

 $<sup>\</sup>star$ Concentration increase from potable water to secondary effluent

- sea water has 35-50 times more dissolved salts than secondary effluent
- secondary effluent contains appreciable quantities of organics, ammonia, and phosphates that are present only in trace amounts in sea water.

It should be noted that in a total recycle process the concentration of inorganic materials in the secondary effluent would be less than the amounts shown in Table 1. The figures in Table 1 are based on a potable supply containing some dissolved inorganic materials. If all potable water in a system were produced by evaporation, the level of dissolved materials in the water would be very low and the treated waste from that system would contain essentially only the increment of dissolved materials added during one cycle from potable water to treated wastewater. Typical increases are shown in Table 1. Obviously, if any portion of the potable supply were produced by some means that did not provide complete demineralization, the dissolved solids content of the treated wastewater would be increased accordingly.

These differences in sea water and secondary effluent should markedly affect their relative desirability as evaporator feedwater. In the case of sea water the very high dissolved salt content causes problems with scaling, corrosion, and boiling point elevation. 4,5 Scaling is controlled by pretreatment of feedwater and limitation of maximum temperature to 250°F. Since heat transfer coefficients increase with temperature, the 250°F limitation on sea water evaporators significantly affects the overall economics. The high salt content also affects overall economics by requiring certain high cost materials of construction. The overall economic effects of boiling point rise

are significant also and usually dictate that concentration beyond 7% solids (recovery of half the feedwater) is uneconomical.

By comparison, the maximum operating temperature for secondary effluent should be considerably above 250°F since the concentrations of scale forming materials are considerably lower in this water. Materials of construction for a secondary effluent evaporation plant would likely be different and possibly less expensive than for a sea water plant. Copper alloys, common in sea water conversion plants, probably would be unsuitable because of possible ammonia attack. 4,6,7 The effects of boiling point rise would be negligible by comparison since 97-98% of a secondary effluent feed could be recovered before the solids concentration in the remaining 2-3% reached the 35,000 ppm level of sea water. Hence, with regard to the effects of inorganic salts, secondary effluent has a very clear advantage over sea water.

Organics in secondary effluent make this a less desirable feed for evaporation since many of them are volatile and would be carried over with the steam into the product water. These volatile organics are expected to create taste and odor problems in the product water. Ammonia, likewise, is expected to carry over unless the pH is kept low and ammonia would be an undesirable addition to the product water. Regarding scaling, organics could be either very detrimental or possibly helpful. Obviously, if organic fouling occurred, heat transfer would be greatly lowered. However, it is possible that the organics could act as a dispersing agent tending to keep insolubles from coating the tube walls. Also, a light film of organics on heat exchanger surfaces could promote drop-wise condensation and thus increase heat transfer. Only actual operation under representative conditions could determine

the full effects of organics in secondary effluent.

Phosphates should be advantageous in that they will precipitate calcium as calcium phosphate which normally does not form scale-like deposits. The dispersive actions of organics in the liquid phase may serve to keep the calcium phosphate suspended in solution.

Overall, it appears that the potential economic advantages of secondary effluent over sea water for fresh water production may more than offset the disadvantages. Certainly this possibility seems likely enough to justify further research..

# Previous Work on Evaporation of Treated Sewage

Previous work on evaporation of sewage treatment plant effluent has been limited to several desk top evaluations using presumably reasonable assumptions and four studies that involved actual experimental work.

In 1960 K. C. D. Hickman was commissioned by the Robert A. Taft Sanitary Engineering Center to compare distillation with other unit processes for the reclamation of wastewaters. Hickman concluded that the relatively high cost of distillation (estimated at \$1.00/1,000 gal.) should limit it to processing only part of the water in a total recycle scheme. Other less expensive treatment procedures should be used for the remaining parts of the system. Hickman reported the results of one experimental run using treated sewage from a Rochester, N. Y., sewage disposal plant. The specific evaporator used was not described but operating conditions were reported as 120°F (1.69 psia). In the author's opinion, "the physical properties, flavor, and odor of the distillate were good, and that with mild chlorination, or equivalent, the distillate would be potable and acceptable for municipal reuse."

Possible contamination of the distillate samples prevented any valid conclusions based on actual analysis.

In 1963 an economic analysis and a pilot plant study of wastewater distillation were made by J. H. Neale 4 of W. L. Badger Associates. Neale proposed that distillation be used in parallel with some other form of treatment and that the fraction of the total stream subject to distillation be the minimum required to prevent build up of inorganics in the overall system. Analysis of data from 22 cities indicated that the fraction of the total stream that would have to be distilled ranged from 23.6% to 63.7% and averaged 42.7%. A pilot plant study was carried out in a long tube vertical (LTV) evaporator utilizing a single tube 14 feet long. The primary objective of the tests was to evaluate tube fouling and no provision was made to separate and collect product water. Using primary effluent and no pretreatment. tube fouling was noted after four days. Operation was at 212°F. A second seven day run at 212°F was made using secondary effluent with no pretreatment. Again tube fouling was noted. The effervescence noted when acid was added to this scale led Neale to conclude that it was largely calcium carbonate. Subsequent runs were made using secondary effluent treated with inhibited hydrochloric acid for pH adjustment. Feed pH was maintained at less than 5.7 most of the time. An eight day run at 212°F and a twelve day run at 234°F were made with no scaling problems. Neale concluded "that waste treatment by distillation will probably be applicable to renovation of wastewater at temperatures equal to or exceeding those used for sea water conversion, and that raw sewage, after removal of most of the suspended matter, may be used for distillation plant feed."

In June, 1963, American Machine and Foundry, Inc. obtained an Advanced Waste Treatment Research contract from the government to conduct research on flash evaporation of treated sewage. 10 The equipment consisted of an eight stage flash unit. The still body was steel with titanium heat transfer surfaces. Due to operating problems, very limited testing was carried out. Using secondary effluent under relatively high temperature conditions, 250-300°F, severe fouling of the preheater and some fouling of the feed side of the condenser tubes occurred. The scale appeared to be largely organic. The product water had a strong, disagreeable odor. However, treatment with activated carbon removed the odor. 11-13

In 1965, O'Connor  $^{8}$  and co-workers at the Robert A. Taft Sanitary Engineering Center in Cincinnati, Ohio, conducted laboratory scale tests on distillation of municipal effluents. Batch tests were made using primary effluent, trickling filter effluent, and extended aeration effluent. Temperatures used were 158, 212, and 338°F and pH ranged from 3 to 11. At the lower pH values it was hoped that the ammonia would be retained in the concentrated liquid. At the higher pH values, hopefully all the ammonia would distill over in the first small fraction of product. All feed samples were filtered prior to evaporation to remove solid particles larger than about 5 microns. It was found that ammonia was present in the product water for all feeds with pH above about 3.5. With feeds containing around 7-8 mg/1 $\mathrm{NH_{3}\text{-}N}\text{,}$  ammonia in concentrations above 1.0 mg/l  $\mathrm{NH_{3}\text{-}N}$  typically was found in the first 40--50% of the distillate. Even at pH 11, ammonia in the concentrations above 1.0  $\mathrm{mg/1~NH_{3}\text{--}N}$  was found in at least the first 30% of the distillate. Hence it appeared impractical to

try, on a large scale, either to eliminate ammonia from the distillate by lowering the feed pH or to concentrate all the ammonia in the first small fraction of the distillate by raising the feed pH.

Tests with extended aeration plant effluent having ammonia concentrations less than 2.2 mg/l  $\rm NH_3$ -N showed that, at feed pH values under 5.5, all product fractions had ammonia concentrations less than 1.0 mg/l  $\rm NH_3$ -N. No carry over of nitrate was observed.

Odors were present in all products and scemed to be more
"prominent and enduring" at high temperatures. Product odor seemed
to be independent of feed pH. Powdered carbon at 1 gm/l removed all
odors from products produced under vacuum and atmospheric conditions.
However, carbon treatment only partially removed the odors from the
products made at high temperature and pressure conditions.

Economic analyses of distillation of treated wastewaters have been made by several people. Gerster 14 has made a desk top study of multistage flash, multiple-effect, and recompression-flash evaporation with respect to how these processes might apply to wastewaters. He concluded that "For all types of equipment the cost for the distillation step alone is somewhat less than for sea water, but inclusion of costs for feed pretreatment and ultimate disposal of blowdown, bring the cost up to about that for sea water."

Stephan<sup>15,16</sup> in 1965 predicted that wastewater evaporation should cost about the same as sea water evaporation. Also, he concluded that a parallel renovation system utilizing evaporation on half of the recycle stream would cost about the same as using electrodialysis on the entire stream.

# Objectives of this Research

After reviewing the previous work on evaporation of sewage treatment plant effluent, it appears that additional information is needed in several areas. First, very little work has been done on a continuous basis utilizing equipment simulating full scale operations. Also, renovation by evaporation should be considered in relationship to any additional pretreatment or post treatment processes that may be required to render the water acceptable for reuse.

Based on these needs, the following objectives were set for this research.

- To define the relationships between product water quality, feedwater quality, and evaporation conditions.
- To define the relationships between post evaporation polishing required, feedwater quality, and evaporation conditions.
- To define the relationships between evaporator tube scaling, feedwater quality, and evaporation conditions.

#### II. THEORETICAL BACKGROUND

# Separation by Evaporation

Separation of two or more components in a liquid phase is possible by evaporation and subsequent condensation of the vapor if the relative concentrations of materials in the vapor are different than the relative concentrations in the original liquid state. In the case of sea water conversion, the salt concentration in the original liquid is about 35,000 ppm but is essentially zero in the vapor state. The behavior of multicomponent systems can be described theoretically by Raoult's law or Henry's law.  $^{17,18}$  Unfortunately, for most solutions, neither of these laws can be applied over the complete range of concentrations. Raoult's law is generally applied to the solvent or predominent component in a liquid system. The law states that the partial pressure of component a,  $P_a$ , is equal to the mole fraction of a in the solution,  $X_a$ , times the vapor pressure of pure a at that same temperature,  $P_a^0$ .

$$P_a = X_a P_a^0 \tag{1}$$

Henry's law generally applies only to solutes in dilute concentrations and states that the partial pressure of b,  $P_b$ , is equal to the mole fraction of b in the solution,  $X_b$ , times a constant, k, termed Henry's law constant.

$$P_b = X_b k \tag{2}$$

The greatest practical application of these laws is in very

dilute solutions. As  $X_a \longrightarrow 1$  Raoult's law becomes more exact and as  $X_b \longrightarrow 0$  Henry's law becomes more accurate. Hence, both can be accurately termed limiting laws. Also, under ideal conditions, i.e. where the intermolecular forces between molecules of A, B, and A and B are all equal, the Henry's law constant, k, becomes  $P_b^o$  and Henry's law and Raoult's law are identical.

### Volatile Organics

Unlike sea water, treated sewage does contain materials that can be classified as volatile under temperatures likely to be encountered in evaporation. These include numerous organic compounds and ammonia. Since in all cases we are talking about very low concentrations, we can assume that Henry's law will apply to the solutes. Unfortunately, the limited work that has been done to characterize sewage treatment plant effluent plus the day-to-day variations in effluent quality make it virtually impossible to construct a predictive model for specific compounds in the effluent. 19,20 Also, k values are not available for all the organic compounds that may be present. To complicate matters further, it appears that some of the organics in the evaporator feed are broken down by heat in the evaporation process to produce smaller, more volatile compounds. 8 This appears to be particularly true as evaporator temperature is increased. Therefore it is impossible to predict either the amount or the specific compounds one might expect to carry over during evaporation.

Nevertheless, some volatile organic material in the evaporator feed can be expected to carry over into the product water. Exactly how much is carried over will be dependent on the relative volatility of the compounds and the evaporator operating conditions. The effect

of these compounds in the product water will be to increase the COD and probably to add some taste and odor.

### Ammonia

Nitrogen in the form of ammonia is found in the effluent from almost all biological treatment processes. The exception is treatment involving extended oxidation conditions wherein the ammonia is oxidized to nitrate. Even in the extended aeration processes it is not unusual to find 0.5-1.0 mg/l  $NH_3$ -N.

The 1962 Public Health Service Drinking Water Standards 21 set no limit on ammonia concentration. Likewise the more stringent Water Quality  $\operatorname{Goals}^{22,23}$  established by the American Water Works Association make no mention of ammonia. In a 1968 report issued by the Federal Water Pollution Control Administration on raw water quality criteria for public supplies, a permissible criteria of 0.5 mg/l  $\mathrm{NH_{3}\text{-}N}$  and a desirable criteria of less than 0.01 mg/1 NH2-N were recommended.24 The rationale for these low limits was given as follows. "Ammonia is a significant pollutant in raw water for public water supplies because its reactions with chlorine result in compounds with markedly less disinfecting efficiency than free chlorine. In addition, it is frequently an indicator of recent sewage pollution."24 In order to satisfy a chlorine demand of 1 mg/1  $\mathrm{NH_{3}}$ -N and produce a free chlorine excess, i.e. "breakpoint" chlorination, about 10 mg/l of chlorine are required.<sup>8</sup> Hence, for economic reasons alone, it appears undesirable to have ammonia above 1.0 mg/l in the final product water. The fact that no standard for ammonia in potable water exists probably reflects that historically ammonia has not been a problem and not that ammonia was not considered undesirable.

The volatilization of ammonia in an ammonia-water system is influenced by several factors, pH, ammonia concentration, and solution temperature. Ammonia dissolves in water to form NH<sub>4</sub>OH which is in equilibrium with ammonium ion as follows.

$$NH_3 + H_2O \stackrel{\longrightarrow}{=} NH_4^+ + OH^-$$
 (3)

$$K_{b} = \frac{\left[NH_{4}^{+}\right]\left[OH^{-}\right]}{\left[NH_{3}\right]} \tag{4}$$

If values of  $K_{\overline{b}}$  at various temperatures are known, the relative concentrations of NH $_4^+$  and NH $_3$  can be calculated as a function of pOH. Now, since

$$K_{\mathbf{w}} = \left[ \mathbf{OH}^{-} \right] \left[ \mathbf{H}^{+} \right] \tag{5}$$

and values of K  $_{\rm W}$  at various temperatures are known, the relative concentrations of NH  $_4^+$  and NH  $_3$  as a function of pH can be determined.

$$\begin{bmatrix} H^{+} \end{bmatrix} = \frac{\begin{bmatrix} NH_{4}^{+} \end{bmatrix} K_{W}}{\begin{bmatrix} NH_{3} \end{bmatrix} K_{b}}$$
 (6)

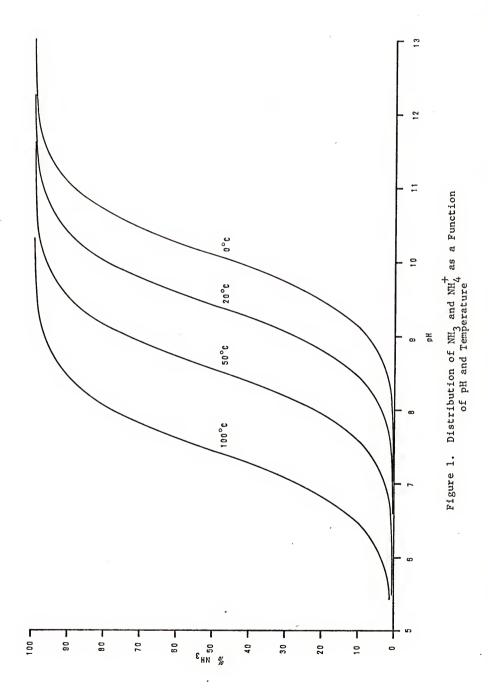
$$pH = log \frac{NH_3}{NH_4^+} + pK_w - pK_b$$
 (7)

Values of  $pK_w^{25,26}$  and  $pK_b^{27,28}$  for ammonia are shown in Table 2. Using these values and equation 7, the distribution of  $NH_3$  and  $NH_4^+$  was calculated as a fraction of pH at various temperatures. The results are in excellent agreement with similar data published in an American Water Works Association committee report on nutrients in water. The source of their data was not given. Figure 1 shows that at  $20^{\circ}$ C and pH 7 more than 99% of the ammonia is present as  $NH_4^+$  and hence not subject to evaporation. However, as the temperature

TABLE 2

DISASSOCIATION CONSTANTS FOR AQUEOUS AMMONIA AND WATER

Temp.	pK for water25,26	pK for Aqueous bAmmonia27,28
0	14.9435	4.862
10	14.5346	4.804
20	14.1669	4.767
25	13.9965	4.751
30	13.8330	4.740
40	13.5348	4.730
50	13.2617	4.723
60	13.0171	·
100	12.30	4.87



increases, the equilibrium shifts and there is more ammonia available for evaporation. This would explain the fact that 0'Connor  $^8$  found that he had to lower the feed pH to under 5 in order to eliminate ammonia in the product water.

The relative volatility of ammonia as compared to water strongly influences the relative amounts of ammonia and water in the liquid and gas phases during evaporation. At the very low ammonia concentrations encountered in treatment plant effluents Henry's law should apply. Unfortunately, no data have been published for ammonia-water systems in the 0-20 mg/1 NH<sub>2</sub>-N concentration range. However, considerable data are available for ammonia-water systems in ammonia concentrations above 1% (10,000 ppm). 30-32 These data show the relative concentration of ammonia in the vapor phase to be much higher than in the liquid phase. This concentration factor increases as ammonia concentration in the liquid decreases and is around 10 at the 1% (ammonia in liquid) level. Therefore, at the much lower ammonia concentration levels we are considering, the ammonia concentration factor from liquid to gas should substantially exceed 10. Concentration of ammonia by evaporation and condensation is well recognized and is an accepted laboratory procedure in the analysis of ammonia. 33

The effect of temperature on ammonia volatilization from an ammonia-water system is that the ammonia concentration factor decreases slightly as temperature increases. <sup>30</sup> However, this effect does not appear to be significant over the temperature range that would be considered for evaporator operations.

From a theoretical standpoint, it appears that ammonia carry over to the products is dependent on the pH of the feed solution and

the evaporation temperature. Since the ammonia-ammonium ion equilibrium shifts in favor of ammonia as temperature increases, one might well expect to find ammonia in the product water at pH values at least as low as 6 if evaporation is occurring under pressures exceeding atmospheric.

# Scaling and Fouling

Loss of heat transfer efficiency may result from inorganic scaling, organic fouling or a combination of the two.

Inorganic scaling occurs when the solubility of the scaling compound is exceeded. This may occur because of temperature increase, since the solubilities of many scale forming salts decrease with increasing temperature. In an evaporator, the continual evaporation of water concentrates the non-volatile ions in solution to the point that the entire solution may be saturated with respect to a particular salt. However, scaling may occur at localized points in the system even though the solubility of the particular scaling salt is not exceeded in the bulk of the water. This can occur because the thin film of liquid immediately adjacent to the heating surface tends to become more saturated than the remaining water.

In the first case, where the entire solution becomes supersaturated, precipitation may occur in the solution. This precipitate may remain suspended in the solution and be removed with the concentrated effluent. However, it may settle on the tube surfaces and "bake" in place as a scale. The second case is more detrimental in that the precipitation tends to occur directly on the heating surface and there is little chance that the solid thus formed will be suspended in the solution. 34,35

With regard to boiler scaling, the Betz Handbook makes the following comments.

It is well to realize that the prevention of boiler scale cannot be predicted by any basic chemical principal. It is the physical characteristics of the precipitate formed in the boiler water that determines whether or not the precipitate will tend to tightly adhere to the boiler heating surfaces—the chemical characteristics of the precipitate are relatively unimportant. Thus, from a chemical standpoint sodium silicate will precipitate the calcium and magnesium salts just as well as a phosphate or carbonate. However, it is the physical characteristics of the precipitate that are of importance, for a precipitate of calcium silicate will tightly adhere to the heating surfaces. 34

Treatment for prevention of inorganic scale may be external or internal. External treatment involves removing the potential scale forming materials from the water prior to its introduction into the evaporator, boiler, etc. Internal treatment involves the selective precipitation of the potential scale forming materials in the form of non-scaling sludges. This may include the addition of materials designed to keep the sludges fluid and in suspension so that they can be removed from the boiler or evaporator in the blowdown or concentrated effluent.

Calcium carbonate is one of the least soluble salts of magnesium or calcium that is likely to occur in wastewater evaporation. At temperatures below boiling this scale can be controlled by adjusting the carbonate equilibrium system in favor of  ${\rm H_2CO_3}$  and  ${\rm HCO_3}$  by pH control. Excellent discussions of this equilibrium system have been

presented by Langelier,  $^{37,38}$  Dye,  $^{39,40}$  and Weber and Stumm.  $^{41}$  However, pH control of the carbonate system cannot be accomplished under boiling conditions where the  $^{C0}_2$  solubility is zero. Under such conditions  $^{20}_3$  converts to  $^{C0}_3$  and  $^{20}_3$ , and  $^{20}_3$  can precipitate according to the following reactions.

$$2HCO_3^- \rightleftharpoons H_2O + CO_2 + CO_3^-$$
 (8)

$$ca^{++} + co_3^{=} \Rightarrow caco_3$$
 (9)

Since  ${\rm CO}_2$  solubility is zero, the continual removal of  ${\rm CO}_2$  from the system forces the complete decomposition of the  ${\rm HCO}_3^-$ .

The obvious way of controlling calcium carbonate scale is by removal of the calcium. This could be done by hot or cold lime-soda softening or by ion exchange. 34-36,42 However, in the case of wastewater evaporation, softening would add significant additional costs to the overall system. Ion exchange is questionable because the effects of organics on fouling of the exchange resin are not known.

Calcium carbonate scale also can be controlled by pH adjustment and degassing prior to the evaporator. This involves lowering the pH to put the carbonate in the  ${\rm H_2CO}_3$  form and then removing it as  ${\rm CO}_2$  by degassing.

Finally, calcium carbonate scaling may be controlled internally by controlled precipitation of the calcium as a non-scaling sludge. 34-36,42 The most common procedure is to precipitate the calcium as tricalcium phosphate or as hydroxyapatite, a mixed calcium phosphate and hydroxide. Several sodium phosphates may be used including trisodium phosphate, disodium phosphate, sodium metaphosphate, and monosodium phosphate. 34 In some cases it is necessary to add organic dispersants to prevent

the sludge from growing into aggregates. Typical types of organic materials used for this purpose are tannins, lignins, glucose derivatives, and starches. 34,35,42

Precipitation of calcium by phosphate and dispersal of the sludge by organics has promise for use in wastewater evaporation since both phosphates and organics naturally occur in this water. However, individual wastewater streams may or may not contain sufficient phosphate and the dispersal abilities of the organics in wastewater are not known.

# Calcium Sulfate

Calcium sulfate presents a more serious problem than calcium carbonate because it forms a very hard and adherent scale. 36,42

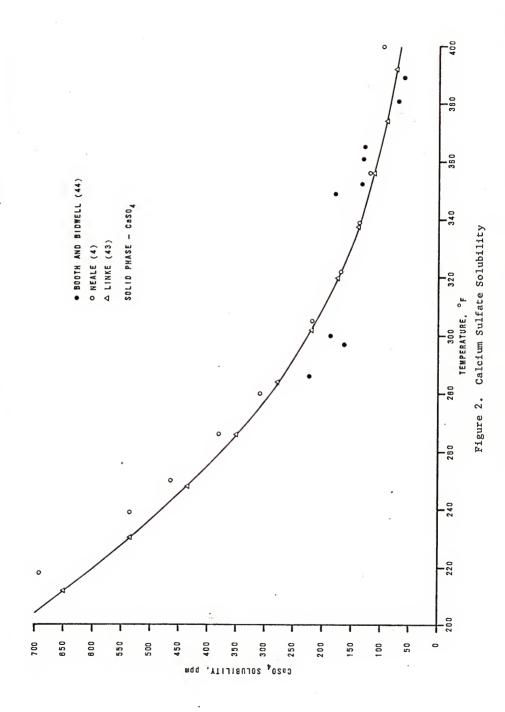
Calcium sulfate solubility decreases with temperature as shown in Figure 2.43,44

It should be noted that this is the solubility of pure calcium sulfate, anhydrite, in pure water. In wastewater, calcium sulfate will have a higher solubility due to the increase in ionic strength caused by the other ions in solution.

Control of calcium sulfate scale is accomplished in much the same way as control of calcium carbonate. External treatment to remove or reduce the calcium content of the water is applicable. 34-36,42 Internal treatment to remove calcium by controlled precipitation with phosphate can likewise be used. 34-36,42 The comments made previously regarding these control measures all apply here.

# Magnesium Hydroxide

Magnesium hydroxide has a solubility of about 5 ppm at  $212^{\circ}F$  decreasing to slightly less than 1 ppm at  $392^{\circ}F$ . Control of this scale may be external by removal of the magnesium by softening or



ion exchange. 34-36,42 Internal control can be accomplished by controlled precipitation with phosphate. However, magnesium phosphate tends to form a sticky deposit and requires mechanical removal. 34 Finally, since magnesium forms a hydroxide scale, it can be controlled by maintaining the pH and hence hydroxide concentrations at a low enough level so as to not exceed the solubility.

# Silica

Silica tends to form complex scales which may include calcium, magnesium, aluminum, or may be composed almost entirely of silica. 34,36 These scales are usually very hard, glassy, and adherent. McCoy 42 gives a maximum permissible concentration of silica of 250 ppm at 388°F for boilers. This figure drops to 175 ppm at 421°F. Presumably it increases for temperatures below 388°F.

In some cases too low silica concentrations can cause problems.<sup>34</sup>

If magnesium concentration is high and phosphate is present, the

magnesium will precipitate a sticky magnesium phosphate sludge. However, if sufficient silica is present, magnesium silicate, which is
easier to handle, will be precipitated.

Control of silica scale may be external by hot lime-soda softening or by coagulation with ferric sulfate.  $^{36,42}$  Ion exchange using a strongly basic anion exchange resin regenerated with sodium hydroxide may also be used.  $^{36}$ 

Internal treatment for silica scale, if any treatment at all is required, appears to be better suited for the wastewater evaporation process. This involves maintaining a small excess of phosphate and a ratio of alkalinity to silica of at least one to one. 36

### Organic Scale

Organic materials may cause heat transfer problems by any of several different mechanisms. Suspended organics may simply deposit on heat transfer surfaces. Oils or greases, which have a tendency to coat metal surfaces, may cause problems. The high temperatures in the evaporator may tend to polymerize some organics into high molecular weight insoluble compounds that could coat tube walls. In the latter stages of evaporation, as concentrations of both organic and inorganic materials increase, the possibility of salting out exists.

Another potential organic fouling problem exists in the early stage preheaters (temperature less than  $130^{\circ}F$ ). This is the problem of bacterial growth. It is possible that this could be adequately controlled by periodic shock chlorination.

In contrast to all the potential problems with organic fouling, there are some possible positive benefits. Certain organics may have a dispersing effect which would tend to keep solids suspended. Also, volatile organic vapors carrying over from one effect to the steam chest of the next effect may promote dropwise condensation on the evaporator tubes. If the organic material coats the condensing surface and renders it non-wettable, condensation will occur dropwise and since dropwise condensation gives higher heat transfer, the overall efficiency of the process would be improved. 45

### Bacteria or Virus Contamination

Since in evaporation of sewage treatment plant effluent it is inevitable that bacteria and virus will be present in the feedwater, a question that must be faced is what are the possibilities of these organisms contaminating the product water. At least three highly unlikely events must occur in order to contaminate the product with live bacteria or virus. First, the organism would have to survive the maximum feed temperature in the evaporator. The forward feed multiple effect evaporator design requires that all feedwater pass from the hot end of the system to the cold end. Hence, all incoming water is heated uniformly to the maximum operating temperature. In the case of sea water this temperature is 250°F. However, as pointed out previously, it is likely that the upper temperature limit for sewage treatment plant effluent will be higher than this. For comparison purposes, milk is pasteurized at 161°F and microbiological laboratories sterilize their equipment in autoclaves at 250°F. Sterilization times as low as 10 minutes are used for small samples. Because of the uniform heat conditions obtained in an evaporator, the somewhat shorter holdup times at the maximum temperature should be at least as efficient as 10 minutes in an autoclave.

The second factor dictating against bacteria or virus contamination in the product is the basic separation mechanism being used, i.e. evaporation and condensation. Since bacteria and virus are non-volatile, they cannot contaminate the product by evaporating and condensing with the product. To get into the product water it would be necessary for any organisms to be physically carried over in the vapor stream, i.e. entrainment. The extent to which this would be possible would depend on the type and design of the vapor liquid separation system in each effect.

The third factor that would be used to eliminate any danger from bacteria or virus contamination would be chlorination. As with any municipal potable water supply, all water from the plant would be chlorinated.

Considering these facts, it seems reasonable to conclude that possible danger from bacteria or virus in a forward feed multiple effect evaporator system using sewage treatment plant effluent as feed would be less than the same danger from a conventional coagulation plant using surface sources of raw water.

#### Ultimate Disposal

Practically all wastewater treatment processes involve separation of the incoming liquid into a "clean" and "dirty" stream. In all cases some final disposition must be made of the "dirty" or concentrated waste stream. This problem exists in evaporation of sewage treatment plant effluent. The final concentrated effluent stream from the evaporator probably will be on the order of 3-10% by volume of the incoming stream and contain essentially all the inorganic salts and organic material in the feed stream. Ultimate disposal of the stream will depend on the individual situation; however, several possible means would be ocean outfall, underground disposal, or solar evaporation.

Because of the high temperature anticipated for wastewater evaporation, the concentrated effluent should be completely safe for ocean disposal in a well-designed outfall. Since the effluent would have an appreciable organic content, provision should be made in the outfall design to release the waste far enough offshore so as to preclude the possibility of contamination of beach or estuary areas.

In areas where contamination of ground water would not be a problem, underground disposal of the concentrated effluent could be considered.

Climatic conditions and availability of land would determine

whether or not disposal by solar evaporation would be possible.

Because of the relatively high cost of recovering water by evaporation, it is assumed that this process would not be used if other sources of fresh water were available. In effect, this says that wastewater evaporation will be limited to very arid areas or certain island or coastal areas where little or no fresh water is available. Hence, there would be no possibility of dumping the concentrated effluent into an existing fresh water stream large enough to assimilate the waste.

Finally, it should be noted that ultimate waste disposal problems in an area would not be increased by evaporation of sewage treatment plant effluent. If evaporation were not used, the more dilute waste containing the same absolute amounts of impurities would still have to be disposed of. Therefore, it is likely that evaporation would reduce rather than add to ultimate disposal problems.

#### III. RESEARCH PLAN

The objectives of this project, particularly with regard to scaling evaluations, dictated that 1) the work be done in equipment of sufficient size to simulate full scale operation, 2) operation be continuous rather than batch, and 3) various effluents typical of what could be expected from municipal waste treatment plants be used as evaporator feed.

Within these constraints the following approaches were taken to various parts of the overall research plan.

#### Experimental Equipment

The long tube vertical (LTV) evaporator design was selected for use in this work. In discussing all types of evaporators, Standiford 47 points out that more evaporation is accomplished in LTV evaporators than any other type and makes the following comments regarding the LTV design.

The widespread use of the LTV evaporator is due partly to the ability to build large single units, partly because of the high heat-transfer performance exhibited under most conditions, and partly because of the simplicity and hence cheapness of construction (simply a shell and tube heat exchanger surmounted by a vaporliquid separator).

Because of their high capacities, and lower costs than for any other type, rising-film LTV evaporators are used whenever possible. . . . While they cannot usually handle crystallizing solutions, they are widely used for viscous and mildly scaling liquors. They are well suited to corrosive solutions because heat-transfer coefficients are generally high requiring a minimum of expensive heating surface, and tube replacement is simple. . . . The principal disadvantage of the rising-film LTV evaporator is the poor heat-transfer performance at low temperature-differences or at low temperatures.

For these reasons and because the rising-film LTV design is readily adaptable to pilot scale size units, this type evaporator was selected for use in this research. Consideration was given to the use of a single effect unit. It was concluded that since any full scale plant would be multiple effect, pilot plant data from a multiple effect unit would be far more valuable than data from a unit that would only simulate the first stage of a multiple effect system.

Since corrosion was not to be studied in this research, it was deemed desirable to construct the evaporator of 316 stainless steel to minimize the possibility of product contamination by corrosion.

As with the evaporator, the accessory equipment used for feed preparation and product polishing was designed to operate continuously.

The entire system was designed by the author. In design of the evaporator provision was made to allow operation as a single stage unit or to operate all three effects in series. In either mode of operation any pressure range from about 28 inches Hg vacuum to 100 psig could be selected. Unfortunately, steam pressure available at the site limited the upper pressure to 50-55 psig.

The evaporator was constructed by Indian River Construction Co., Jacksonville, Florida in cooperation with Reynolds, Smith, and Hills, Architects and Engineers, Jacksonville, Florida.

## Effluents Available

The experimental evaporator was set up adjacent to the 2 MGD Campus Sewage Treatment Plant operated by the University of Florida. This plant handles the flow from the main campus, the teaching hospital and medical center complex, fraternities, sororities, dormitories, an elementary school, and several married student

apartment complexes. Flow from all these sources results in a waste reasonably typical of municipal sewage. The Campus Plant operates a primary treatment unit, a high rate trickling filter, a standard rate trickling filter, and a contact stabilization activated sludge unit. A 9,000 GPD extended aeration unit is also available for use but is not operated routinely. Effluents from any of these units are available for experimental purposes.

#### Experimental Plan

The experimental work was divided into two principal phases. The first phase was directed towards defining the relationships between feedwater quality, product quality, and evaporation conditions. Experimental runs were to be made operating the evaporator as a single effect unit and using progressively poorer quality effluents as feedwater. By this procedure we would minimize the possibility of having major difficulties with excessive organic fouling as was experienced during the work done at American Machine and Foundry, Inc. 10-13 Thus testing was to proceed using, in order, extended aeration plant effluent, contact stabilization plant effluent, high rate trickling filter effluent, and primary effluent until either operational difficulties or product water quality dictated that further testing with poorer quality effluents was not justified. Evaporator operating conditions were to be varied over the range from about 28 inches Hg vacuum to about 50 psig. Also, the pH of the feed was to be varied to see the effects on ammonia carry over to the product. In addition to measuring the relationships between these variables and product water quality, the first testing phase included the evaluation of several post treatment processes. These included activated carbon

treatment, aeration, and ammonia removal by ion exchange.

After completion of the first phase of the experimental work some preliminary judgements were to be made regarding the practical limits for both minimum feedwater quality and maximum evaporator operating temperature consistant with acceptable product water. Using conditions at or near these limits, extended continuous runs utilizing all three evaporator effects were to be made to evaluate scaling or fouling. It was anticipated that these runs would last one to two weeks each. Scaling was to be evaluated both directly and indirectly. By analysis of the overall heat transfer coefficients, Uo, for each stage we would be able to measure scaling by drop in Uo. Also, after each run we planned to clean the evaporator mechanically and measure the amount of scale removed. Also, we planned to analyze the scale to determine the cause of the scaling.

In addition to providing data on scaling, the extended runs would provide considerable additional data on the quality of product that could be expected under the conditions we felt would be most likely to be used in full scale operations.

## Analytical Plan and Procedures

In the analytical work we were concerned with three types of waters: 1) sewage treatment plant effluent or evaporator feedwater,
2) evaporator product water, and 3) the concentrated effluent from the evaporator, and with one solid material, evaporator scale.

Table 3 lists the analyses made on the liquid samples and the procedure used. The very large number of samples involved dictated that automated analysis techniques be used as much as possible.

Chemical Oxygen Demand, COD, was used as an indicator of the

TABLE 3

ANALYTICAL TESTS ON LIQUID SAMPLES

Test	Procedure
COD	Alternate Procedure for Dilute Samples, Standard Methods for the Examination of Water and Wastewater <sup>33</sup>
Ammonia Nitrogen	Industrial Method Ind19-69w of Technicon Corp. Used Technicon AutoAnalyzer
Nitrate Nitrogen	Method of Kahn and Brezenski modified for Technicon AutoAnalyzer <sup>48</sup>
рН	Corning Model 12 pH meter
Odor	Subjective opinion of operator
Total Dissolved Solids	Residue on Evaporation at $103^{\circ}\mathrm{C}$ , Standard Methods for the Examination of Water and Wastewater $^{33}$

organic content of the samples. We anticipated that the primary problem here would be accurately measuring COD on the product samples. In order to strengthen our ability in this area, we spent considerable time over a period of several months trying to perfect a total organic carbon procedure that would be accurate in the 0-3 mg/l range. Unfortunately, with the equipment we had available, we were never able to develop a procedure as precise as the dilute COD procedure and this effort was finally abandoned.

Ammonia nitrogen was one of the most important variables in this study and its significance has been discussed previously.

Nitrate nitrogen was used to indicate the degree of nitrification in the various sewage treatment plant effluents used. Another very significant use of nitrate results was to measure the degree of physical carry over of material into the evaporator product water. Since nitrate is non-volatile, a material balance over the evaporator based on nitrate would be a very accurate measure of physical carry over.

The importance of pH in ammonia distribution and in scaling has been discussed previously. Measurements were made primarily on feedwater samples. Samples were adjusted to  $25^{\circ}C \pm 5^{\circ}C$  prior to measurement.

Odor was subjectively measured by the operating personnel at the time the products were produced. The difficulties involved in obtaining an odor-free room and an experienced panel dictated against more rigorous odor testing.

Total dissolved solids were measured on a few of the feedwaters and concentrated effluent samples, primarily to better characterize

the evaporator feedwater.

Some additional test results taken from the records of the Campus Sewage Treatment Plant were used to characterize influent and effluent materials from the various plant processes.

The scale removed from the evaporator was first dried at  $103^{\circ}\text{C}$  and weighed to obtain a total weight figure. The samples were then fired at  $600^{\circ}\text{C}$  to remove the organic material. The fired residues were dissolved in dilute hydrochloric acid and the solution analyzed as shown in Table 4.

TABLE 4

ANALYTICAL TESTS ON SCALE

Test For	Procedure Used
Ca	EDTA Titrimetric Method, Standard Methods for the Examination of Water and Wastewater <sup>33</sup>
Mg	Atomic Absorption Method Using Beckman DB-G Spectrophotometer
Na	Same as above
Fe	Phenanthroline Method, Standard Methods for the Examination of Water and Wastewater $^{33}$
so <sub>4</sub>	Turbidimetric Method, Standard Methods for the Examination of Water and Wastewater $^{33}$
PO <sub>4</sub>	Method of Murphy and Riley 49

Note: Fired scale samples were dissolved in 0.5N  $\mbox{HC1}$ 

#### IV. DESCRIPTION OF EQUIPMENT

A general flowsheet for the process is shown in Figure 3. Feedwater for the system could come from either an extended aeration plant, a trickling filter plant, or a contact stabilization plant. Before any of these effluents were fed into the evaporator, the dissolved gases and carbonates were removed. This was accomplished by adjustment of the pH and degassing under a vacuum. After degassing, the liquid was fed into a three effect, long tube vertical (LTV) evaporator. This evaporator was built almost entirely of type 316 stainless steel. After evaporation, provision was made to treat the product water either in an activated carbon column or an ammonia selective ion exchange column or both.

#### Degasser

A diagram of the degassing equipment is shown in Figure 4. Effluent from the various sources was put into a 175 gallon agitated tank for mixing and pH adjustment. The agitator was a flat blade 30" in diameter by 1 3/8" high and was driven at 100 rpm. From the mix tank, the effluent passed through a packed column under vacuum to remove dissolved gases. The packed column was 4" 0.D. glass tubing. Vapor and dissolved gases were removed from the column through a condenser and liquid trap by a sliding vane type vacuum pump (Gast Manufacturing Corp. No. 0765-V39). Degassed liquid was pumped from the bottom of the column to one of two 55 gallon open top feed drums by a variable speed screw type pump (Robbins & Myers, Moyno Model 1L4 Type CDQ).

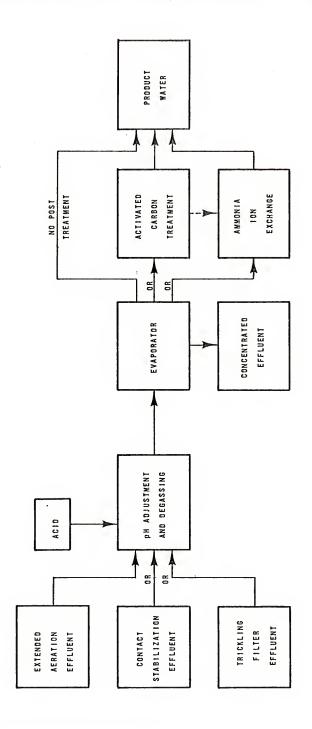


Figure 3. Overall Flowsheet

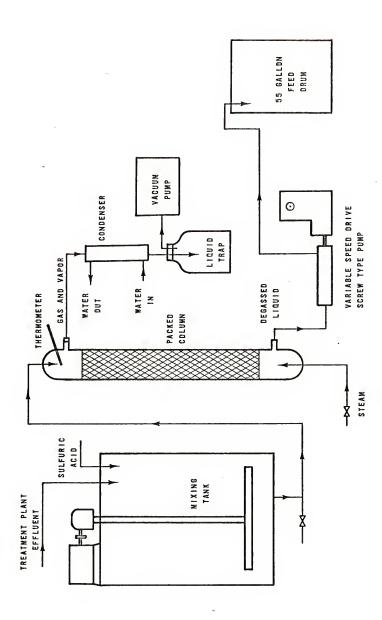


Figure 4. Degassing System Flowsheet

The two feed drums were fitted with polyethylene drum liners. The mixing tank, piping, and pump in the degassing system were of steel construction.

Temperature in the column was measured by a thermometer inserted into the column through a small port. Provision was made to introduce steam into the bottom of the column.

#### Evaporator

A diagram of the evaporator is shown in Figure 5 and a picture of the evaporator is shown in Figure 6. The evaporator was designed to operate either as a triple effect unit or as a single stage unit (3rd effect only). Possible operating conditions in the evaporator ranged from about 28 inches of Hg vacuum to 100 psig. Actual operating conditions under pressure conditions were limited to about 55 psig by the steam pressure available at the site.

Heat for the evaporator could be supplied to either the first or third effect. The heating medium could be either steam or hot water. Steam was taken from the campus steam line and was reduced at the inlet to the evaporator by a steam regulator (Cash-Acme Inc., No. 5681 UE). When operating under high vacuum, it was necessary to reduce the temperature of the heating medium to avoid unrealistically high temperature drops. This was done by using a closed hot water system. The water was circulated continuously through the evaporator and a natural gas fired hot water heater (Odis Stove Manufacturing Co., No. 35DP).served as the heat source.

Feed was pumped into the evaporator by a diaphragm metering pump (Yarway Cyclo/Phram Model No. 071-33-1431). A spring loaded, back pressure valve on the downstream side of the pump prevented liquid

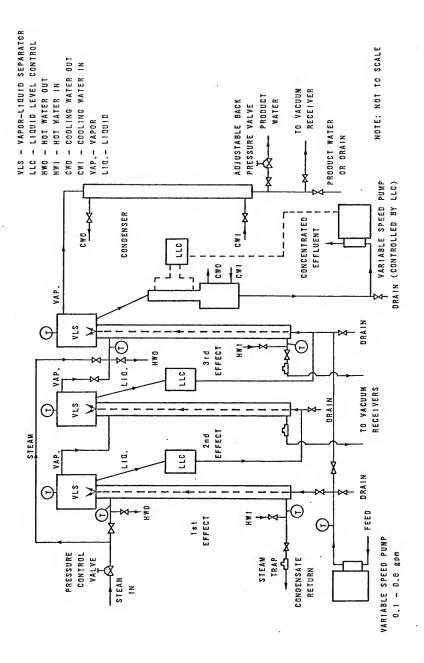


Figure 5. Evaporator Flowsheet



Figure 6. Evaporator

from being drawn through the pump when the system was operating under vacuum conditions. Each of three effects was constructed of 1 inch IPS Schedule 5 pipe, jacketed with 4 inch IPS Schedule 5 pipe. The effective heated length of each effect was 14 feet.

Vapor-liquid separators atop each effect separated the steamliquid mix into two streams. This equipment is shown in Figure 7. The demisters were 316 stainless steel mesh (Otto H. York Co., Style 326). Small vent lines (1/4" O.D. tubing) with needle type control valves connected the steam jacket of each effect with its vapor-liquid separator.

Liquid seals were maintained between stages by float controlled liquid drainers (Armstrong Machine Works, Model No. 21) having 7/32" diameter orifices. These drainers were chrome plated cast iron with stainless steel internals. Originally, drainers with 1/16" diameter orifices were tried, but were replaced because the capacity was marginal and chances of plugging were much greater than with larger units.

To reduce heat loss to the atmosphere, the three evaporator effects and connecting piping were insulated with 1 inch thick asbestos type insulation. The three vapor-liquid separators were completely enclosed with custom built foamglass type insulating covers with aluminum exteriors.

Removal of the concentrated liquid effluent from the last effect was accomplished by a diaphragmmetering pump identical to the feed pump. This pump was automatically controlled (on-off) by liquid level probes located in an enlarged section of the liquid line from the third effect vapor-liquid separator. Cooling coils, located

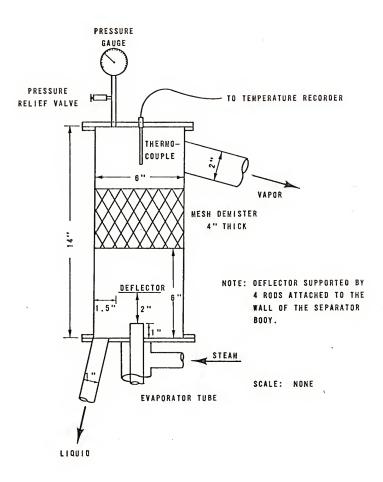


Figure 7. Vapor-Liquid Separator

between the liquid level probes and the pump, cooled the concentrated effluent to prevent flashing of the hot liquid when released to atmospheric pressure. All concentrated effluent piping was steel. The back pressure valve following the pump was a copper alloy.

Condensate was removed from the jackets of the three effects through controlled disc type steam traps (Sarco Company, Type TD-50).

Vapor from the third effect was condensed in a water jacketed condenser. The condenser was a 2" IPS pipe with an effective condensing length of 12 ft.

Product from the three stages was removed by several means, depending on the evaporator operating conditions (See Figures 5 and 8). Under pressure conditions, product No. 3 was removed from the final condenser through an adjustable back pressure valve (Watts Regulator Co., No. 5300A). This valve was of chrome plated copper alloy construction. Product Nos. 1 and 2 were removed through the vacuum receiver system as shown in Figure 8 except that the system was opened at valves D allowing the products to drain continuously to glass carboys.

Under vacuum operation all three products were removed through the vacuum receiver system. Under normal conditions, valves B and C were closed and valves A were open. Valves D were check valves that allowed flow from the upper to the lower tanks only. Hence, product water would drain from the evaporator into the upper tanks and down into the lower tanks through valves D. Vacuum was maintained on the system by a vacuum pump (Zeflex, No. 012-911-1). In order to remove the product water from any one system, solenoid valves B and C were opened and valves A were closed. The product water either drained

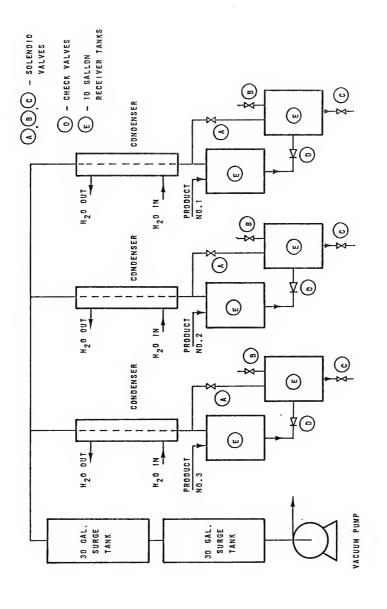


Figure 8. Product Receiver System Flowsheet

through valves C by gravity or was drawn into evacuated carboys. When the lower tank was empty, the cycle was reversed closing valves B and C and opening valves A. Two 30 gallon tanks in the vacuum system provided surge capacity when an air filled tank was cycled back into the system. The instrumentation system provided for automatic cycling of the receiver system to dump product water from each effect on a timed sequence.

All six product receiver tanks were 10 gallon capacity.

Temperature was sensed by copper-constantan thermocouples at various points in the system and automatically recorded by a 16 point recorder (Minneapolis-Honeywell Reg. Co., No. Y153X64P16-X-41(v)). The temperature range of this recorder was 0-350°F. Pressure gauges, installed in the incoming steam line, in each vapor-liquid separator, and in the final condenser, gave continuous visual indication of the pressure at various points in the system.

#### Carbon Column

The carbon column was a 1.94" I.D. pyrex tube four feet long, containing 42" of activated carbon. The column was fed by gravity and flow rate was controlled by a polyethylene needle valve. The activated carbon used was Nuchar WV-W, 12 by 40 mesh, manufactured by West Virginia Pulp and Paper Co.

#### Ammonia Column

The ammonia ion exchange column was a 1.94" I.D. pyrex tube 13" long, containing 10.25" of ion exchange resin. The column was fed by gravity and the flow rate was controlled by a polyethylene needle valve. The ion exchange resin used was natural clinoptilolite, 20 by 50 mesh, from the Hector, California, area.

#### V. EXPERIMENTAL RESULTS

### Initial Testing

As stated in the research plan, the initial testing was designed primarily to define the relationships among evaporator feedwater quality, evaporator operating conditions, and product water quality. Also, to be included was evaluation of several post treatment processes and determination of how they might be included in an overall reuse cycle.

With regards to effluents to be used, it was planned to begin the testing with the highest quality effluent to reduce the likelihood of severely scaling or fouling the evaporator tubes. Testing would then proceed using progressively poorer quality effluents.

The tests on each different effluent were made at three temperature and pressure levels. These conditions are shown in Table 5.

In addition to the temperature variations, the pH values of the feed were varied at each level to examine the effect on ammonia distribution between the concentrated effluent and product.

On selected evaporator products that would be unsuitable for direct reuse, tests were made using activated carbon, ammonia selective ion exchange resins, and aeration to determine the effectiveness of these post treatment processes.

Before any sewage plant effluents were used as evaporator feed, the evaporator was operated using tap water and distilled water. This was done to clean out the inside of the equipment and to develop

TABLE 5

EVAPORATOR OPERATING CONDITIONS FOR INITIAL SINGLE STAGE TESTS

Condition	Evaporating Liquid Temperature and Pressure	Heating Medium Temperature and Pressure
Vacuum	114-120°F 26.5-27" Hg vac.	Hot Water 140-155 <sup>0</sup> F
Atmospheric	212 <sup>0</sup> F 0 psig	Steam 228-238 <sup>0</sup> F 5-9 psig
Pressure	286-288°F 39-41 psig	Steam 300-305 <sup>0</sup> F 52-58 psig

familiarization with the operating characteristics of the equipment. Steam was used to clean the product receiver tanks.

When the evaporator was operated as a single effect unit (3rd stage only), equilibrium conditions for the entire system could be reached in less than 15 minutes. The only exception occurred when the feedwater was changed during a run. When this occurred it could take as long as one hour to completely flush the system and obtain consistant quality product water.

In these initial tests the operating procedure was as follows. The effluent to be used was either pumped or drained by gravity from the sewage treatment plant to the mixing tank. A sample was taken for analysis and the pH of the remaining water was adjusted to the bicarbonate endpoint (pH 4.8-5.0) with 6N sulfuric acid. The water was heated to 120-140°F and then degassed under vacuum. The degassed water was pumped to 55 gallon drums prior to being fed into the evaporator. If required, a pH adjustment of the evaporator feedwater was made at this point using either sulfuric acid or sodium hydroxide.

In the degassing operation there was a 3-5°F drop in temperature caused by flashing as the water entered the top of the column. This corresponded to the measured 0.3-0.5% (of incoming water) evaporation rate from the degasser. The liquid evaporated during degassing usually had a slight odor. However, the odor was normally no stronger than the odor of products produced under atmospheric pressure conditions.

The average degassing rate was 2800 ml/min but ranged from 2100 to 3900 ml/min. This corresponds to an average loading rate in the degassing column of 9.7  $\rm gpm/ft^2$  and a range of 7.2-13.4  $\rm gpm/ft^2$ .

After evaporator start-up, from 30 to 90 minutes were allowed

to attain stable operating conditions and completely flush the system. Product sampling was then begun, usually at 60 minute intervals. Since for each operating condition one drum of feed was required, only one feed sample was taken. One concentrated effluent sample was taken under each set of conditions. Total operating time, once product sampling began, was usually 2 hours, but some runs as long as 5 hours were made.

#### Extended Aeration Plant Effluent

Since extended aeration plant effluent was believed to be the highest quality secondary effluent available, testing was begun using this water. The effluent was from a 9,000 gpd capacity plant built by Chicago Pump. This plant was fed raw sewage that had previously been degritted and ground. Flow rate to the plant was maintained at a constant rate by a constant head and weir arrangement. Excess raw sewage from the Campus Sewage Treatment Plant was pumped to a constant head headbox and the desired amount removed over a V-notch weir. The excess was returned to the Campus Plant. Flow to the plant during the period that the initial batch tests were being made was 6.2 gpm (8,920 gpd), which is the rated capacity of the plant. The plant had been in operation for about four months prior to the initial tests and the mixed liquor suspended solids in the aeration basin had stabilized at about 4,200 mg/1.

Thirteen runs were made using extended aeration plant effluent, five each under atmospheric and pressure conditions and three under vacuum conditions. The results of these runs are shown in Tables 6, 7, and 8.

All products had odors strong enough to eliminate the possibility

TABLE 6

INITIAL TESTS, EXTENDED AERATION EFFLUENT, VACUUM CONDITIONS

		Run No.	
	1	2	3
Operating Conditions			
Feed Rate, ml/min	647	647	632
Evaporation Rate, ml/min	93	95	117
% Evaporated	14.4	14.7	18.5
Hot Water Temp., F	144	144	155
First Stage Temp., F	116	116	122
Feed Temp., F	91-96	98-106	95-103
Feed, Before Degassing			
Alkalinity, mg/1 CaCO3	25	25	32
pH 3	6.5	6.5	6.5
COD, mg/1	49	49	65
$NH_2-N, mg/1$	0.8	0.8	0.1
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	19	19	
Feed, After Degassing			
pН	5.3	5.8	6.6
COD, mg/1	46	47	71
NH <sub>3</sub> -N, mg/1	0.7	0.9	0.1
$NO_3^3-N$ , mg/1	18		
Product			•
рН	6.6	5.9	7.2
COD, mg/1	1.3	2.6	4.5-6.3
NH <sub>3</sub> -N, mg/1	0.04	0.11	<0.1-0.3
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	22		
Concentrated Effluent			
pH	6.1	6.2	7.2
COD, mg/1	55	54	71
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	1.0	1.0	
NO3-N, mg/1	22		

TABLE 7

INITIAL TESTS, EXTENDED AERATION EFFLUENT, ATMOSPHERIC CONDITIONS

		Ru	n No.			
	1	2	3	4	5	
Operating Conditions						
Feed Rate, m1/min	640	630	630	655	640	
Evaporation Rate, m1/min	156	146	177	186	293	
	24.4	23.2	28.1	28.0	45.8	
% Evaporated Steam Temp., oF	226	226	230	229	238	
First Stage Temp., F	212	212	212	212	212	
Feed Temp., F	96-104		88-104		80-82	
Feed, Before Degassing						
Alkalinity, mg/l CaCO3	27	27	34	40	50	
рН	6.5	6.5	6.7	6.3	6.7	
COD, mg/1	23	23	27	57	85	
NH <sub>3</sub> -N, mg/1	1.4	1.4	0.42	0.09	0.42	
NO <sub>3</sub> -N, mg/1	7.0	7.0		9.0	8.3	
Feed, After Degassing						
pН	5.4	5.9	6.1	6.3	6.5	
COD, mg/1	36	32	28	65	74	
NH <sub>2</sub> -N, mg/1	0.35	0.37	0.31	0.08	0.43	
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	7.5	7.3		9.0	8.3	
Product						
pН	5.5	5.7	5.9	6.0	6.1	
COD, mg/1	0.0-0.6	0.5-0.7	2.6-3.2			
NH <sub>2</sub> -N, mg/1	0.05	0.10	0.16	0.03	0.36	
$NO_3^3-N$ , mg/1	0.00	0.00				
Concentrated Effluent						
pН	6.3	6.4	6.5	6.3	6.7	
COD, mg/1	41	40	37	87	137	
NH <sub>3</sub> -N, mg/1	0.44	0.47	1.8	0.10		
$NO_3^3$ -N, mg/1	8.1	8.1		13	14	

TABLE 8

INITIAL TESTS, EXTENDED AERATION EFFLUENT, PRESSURE CONDITIONS

		Ru	ın No.		
	11	2	3	4	5
Operating Conditions					
Feed Rate, ml/min	630	635	625	625	630
Evaporation Rate, m1/min	165	166	146	182	219
% Evaporated	26.2	26.2	23.4	29.1	34.8
Steam Temp., F	299	299	300	302	304
First Stage Temp., F	286	286	288	288	288
Feed Temp., F	96-104	104-114	100-115	98-118	109-120
Feed, Before Degassing					
Alkalinity, mg/l CaCO3			42	57	49
pH 3	6.5	6.5	6.5	6.5	6.7
COD, mg/1	52	52	97	110	85
NHN_ mg/1	1.0	1.0	0.40	1.3	0.42
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	7.7	7.7	9.3	7.4	8.3
Feed, After Degassing					
pН	4.8	5.6	5.9	6.2	6.5
COD, mg/1	47	48	90	94	74
NH <sub>3</sub> -N, mg/1	1.0	0.8	0.33	1.4	0.43
NO3-N, mg/1	7.9	7.7	8.6	7.0	8.3
Product					
pН	5.5	5.5	6.2	6.8	6.3
COD, mg/1	3.1-3.2	2.8	3.0-5.1	5.3-12.8	3.3-5.0
NH <sub>3</sub> -N, mg/1	0.15	0.22	0.30	1.2	0.59
$NO_3^3-N$ , mg/1	0.01	0.02			
Concentrated Effluent					
pН	5.9	6.1	6.5	6.0	6.4
COD, mg/1	56	52 ·	106	104	106
	1.4	1.4	0.43	1.5	0.36
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	8.4	8.5	12	10	12

of direct municipal reuse. These odors ranged from very slight musty for products produced under vacuum to moderately strong fecal for products produced under pressure conditions.

# Contact Stabilization Plant Effluent

Contact stabilization plant effluent was taken from the Campus Sewage Treatment Plant. This unit treats about 0.7 MGD of raw sewage. Performance data for this plant are shown in the appendix.

Effluent from this unit was taken from the final clarifier prior to chlorination. Fourteen runs were made, five each under vacuum and pressure conditions and four under atmospheric conditions. The results of these runs are shown in Tables 9, 10, and 11.

All products had odors sufficiently strong to eliminate the possibility of direct municipal reuse. These odors ranged from musty for products produced under vacuum conditions to a rather strong, disagreeable, fecal type odor for products produced under pressure conditions.

# Trickling Filter Plant Effluent

Trickling filter plant effluent was taken from the high rate trickling filter operated by the Campus Sewage Treatment Plant. This unit treats about 0.25 MGD of raw sewage. Performance data for this plant are shown in the appendix.

Effluent from this unit was taken from the final settling basin prior to chlorination. This effluent was noticeably different from the two effluents used previously in that it had a grey color and a slight odor. Six runs were made, two each under each of the three temperature and pressure conditions. The results of these runs are shown in Tables 12, 13, and 14.

TABLE 9

INITIAL TESTS, CONTACT STABILIZATION EFFLUENT, VACUUM CONDITIONS

	Run No.				
	1	2	3	4 ,	5
Onematica Caulitain					
Operating Conditions	600	605		4	
Feed Rate, ml/min	630	635	625	645	630
Evaporation Rate, ml/min	78	88	90	128	79
% Evaporated	12.4		14.4	19.8	12.5
Hot Water, Temp., oF	140	141	141	146	140
First Stage, Temp., F	114	113	114	113	114
Feed Temp., F	89-98	87-93	91-96	92-108	93-106
F					
eed, Before Degassing					
Alkalinity, mg/1 CaCO3	70	82	82	93	70
pH	6.9	7.1	7.1	7.2	6.9
COD, mg/1	23	23	23	30	23
NH <sub>3</sub> -N, mg/1	5.1	6.5	6.5	9.0	5.1
$NO_3^3$ -N, mg/1		0.9	0.9	0.28	
3 , 0			• • • •	0.20	
Feed, After Degassing					
рН	5.6	5.6	6.3	6.7	7.7
COD, mg/1	29	20	19	35	24
NH <sub>3</sub> -N, mg/1	6.4		6.6	8.5	5.8
NO3-N, mg/1	0.5	0.4	0.5	0.31	5.0
	0.5	0.4	0.5	0.51	
Product					
рН	6.9	6.7	7.4	8.4	9.1
COD, mg/1	3.6				4.0
NH3-N, mg/1	.1.6			2.9	6.9
NO3-N, mg/1	.1.0	0.14		0.00	0.9
103 N, 116/ 1				0.00	
Concentrated Effluent					
pH .	7.4	6.8	7.3	7.4	8.2
COD, mg/1	29	22	22		23
NH <sub>3</sub> -N, mg/1	6.4	7.7	7.3	7.6	
NO <sub>3</sub> -N, mg/1	0.4	0.4	0.5		5.9
1103 11, mg/ 1	0.0	0.4	0.5	0.36	

 $<sup>^{\</sup>rm a}$  This run immediately followed Run No. 5 and the high NH  $_{\rm 3}\text{-N}$  result may have been due to contamination from the previous run.

TABLE 10

INITIAL TESTS, CONTACT STABILIZATION EFFLUENT, ATMOSPHERIC CONDITIONS

		Run No	).	
	1	2	3	4
Operating Conditions				
Feed Rate, ml/min	635	640	630	640
Evaporation Rate, ml/min	169	144	172	163
	26.6	22.5	27.3	25.4
% Evaporated Steam Temp., or	230	227	230	229
First Stage Temp., °F	212	212	212	212
Feed Temp., F	95-104	94-106	97-108	84-108
Feed, Before Degassing				•
Alkalinity, mg/l CaCO <sub>3</sub>		101		93
pH	6.9	7.1	6.9	7.1
COD, mg/1	24	68	24	27
NH <sub>2</sub> -N, mg/1	3.3	6.6	3.3	7.0
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	3.1	0.17	3.1	0.49
Feed, After Degassing				
pН	5.1	5.7	5.9	6.5
COD, mg/1	29	62	25	32
NH <sub>3</sub> -N, mg/l	3.5		4.3	9.2
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	3.1	0.23	3.1	0.48
Product				
рН	5.7	7.1	6.4	6.6
COD, mg/1	4.7-5.0	2.9-3.5		
NH <sub>3</sub> -N, mg/1	0.22	0.9	0.3	4.2
NO3-N, mg/1				0.01
Concentrated Effluent				
pН	6.4	6.4	6.4	7.5
COD, mg/1	30	64	27	32
NH <sub>2</sub> -N, mg/1	4.0		3.7	10.0
$NO_3^3$ -N, mg/1	4.1	0.41	4.3	0.64

 $\begin{array}{c} \text{TABLE 11} \\ \text{INITIAL TESTS, CONTACT STABILIZATION EFFLUENT,} \\ \text{PRESSURE CONDITIONS} \end{array}$ 

		Rui	n No.		
	1	2	3	4	5
Onematine Conditions					
Operating Conditions Feed Rage, ml/min	635	630	630	615	630
n n . 1/ ·	152	160	173		170
Evaporation Rate, ml/min % Evaporated Steam Temp., F	24.0				27.0
% Evaporated O.	299	299	300	301	299
0					
First Stage Temp., F	286	286	287	284	286
Feed Temp., oF	94-103	86-96	102-111	91-104	97-108
Feed, Before Degassing					
Alkalinity, mg/1 CaCO3	91	69	91	88	69
pH	7.0	7.1	7.0	7.0	7.1
COD, mg/1	23	24	23	35	24
	6.7	4.6	6.7		4.6
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	0.1		0.1	0.38	
3 1, 11, 11	0.1		0.1	<b>0.</b> 50	
Feed, After Degassing					
pН	5.2	5.7	6.5	6.6	7.2
COD, mg/1	22	23	22	25	24
NH <sub>2</sub> -N, mg/1	7.5	5.7	7.2	8.1	6.4
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	0.2	0.1	0.2	0.31	1.1
Product	6.3	6.0	6 9	6.6	6.6
pH				4.8-5.2	
COD, mg/1	2.2	4.2-4.7	3.3		9.4
NH <sub>3</sub> -N, mg/1	2.2	4.3	3.3		7.4
$NO_3^3 - N$ , mg/1				0.02	
Concentrated Effluent			•		
pН	6.8	8.3	7.3	6.8	6.8
COD, mg/1	24	28	25	39	27
	8.5	5.3	7.8	8.9	4.0
NON. mg/1					
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	0.3	0.9	0.4		

TABLE 12

INITIAL TESTS, TRICKLING FILTER EFFLUENT, VACUUM CONDITIONS

	Run No.		
	11	2	
Operating Conditions			
Feed Rate, ml/min	645	640	
Evaporation Rate, ml/min	100	97	
% Faranamahad	15.5	15.2	
Hot Water Temp., oF	142	143	
First Stage Temp., F	113	114	
Feed Temp., F	90-95	95-102	
Feed, Before Degassing			
Alkalinity, mg/l CaCO3	39	39	
pH	6.6	6.6	
COD, mg/1	116	116	
NH <sub>2</sub> -N, mg/1	5.4	5.4	
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	15.2	15.2	
Feed, After Degassing			
pН	5.3	5.9	
COD, mg/1	101	127	
NH <sub>3</sub> -N, mg/1	6.0	5.5	
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	13.0	13.4	
Product			
pН	5.8	6.6	
COD, mg/1	2.0	1.4	
NH <sub>3</sub> -N, mg/l	0.1	0.6	
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	0.03	0.00	
Concentrated Effluent			
pΗ	6.2	6.5	
COD, mg/1		123	
NH <sub>3</sub> -N, mg/1	6.4	6.3	
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	13.4	14.2	

TABLE 13

INITIAL TESTS, TRICKLING FILTER EFFLUENT, ATMOSPHERIC CONDITIONS

	Run No.		
	11	2	
Operating Conditions			
Feed Rate, ml/min	645	650	
Evaporation Rate, m1/min	172	172	
	26.6	26.5	
% Evaporated Steam Temp., F	228	228	
First Stage Temp., F	212	212	
Feed Temp., F	90-102	101-111	
Feed, Before Degassing			
Alkalinity, mg/1 CaCO3	50	50	
рН	7.0	7.0	
COD, mg/1	93	93	
NH <sub>2</sub> -N, mg/1	5.1	5.1	
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	11.4	11.4	
Feed, After Degassing			
pН	5.5	6.1	
COD, mg/1	93	93	
$NH_2-N$ , $mg/1$	5.9	5.7	
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	10.0	10.8	
Product			
pН	5.8	6.5	
COD, mg/1	5.8-6.2	6.2-6.6	
NH <sub>2</sub> -N, mg/1	0.9	1.6	
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	0.06	0.02	
Concentrated Effluent			
pН	6.5	7.0	
COD, mg/1	105	93	
	7.2	6.8	
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	12.6	13.2	

TABLE 14

INITIAL TESTS, TRICKLING FILTER EFFLUENT, PRESSURE CONDITIONS

	Run No.	
	1	2
Operating Conditions		
Operating Conditions Feed Rate, ml/min	630	630
Evaporation Rate, ml/min	140	165
% Evaporated	22.2	26.2
Steam Temp., F	298	300
First Stage Temp., oF	286	286
Feed Temp., F	95-104	103-113
Feed, Before Degassing		
Alkalinity, mg/l CaCO <sub>3</sub>	65.7	65.7
рН	7.0	7.0
COD, mg/1	90	90
NH <sub>o</sub> -N, mg/1	7.5	7.5
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	16	16
Feed, After Degassing		
pН	5.5	6.1
COD, mg/1	84	80
$NH_3-N$ , $mg/1$	8.0	8.8
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	9.4	9.4
Product		
pН	6.0	6.3
COD, mg/1	18.5-20.9	19.6-22.6
NH <sub>3</sub> -N, mg/1	2.4	3.7
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	0.28	0.17
Concentrated Effluent		
pН	6.5	6.6
COD, mg/1	75	76
NH <sub>2</sub> -N, mg/1	10.0	10.3
NH <sub>3</sub> -N, mg/1 NO <sub>3</sub> -N, mg/1	11.6	11.8

All products had odors as bad or worse than odors of products produced from extended aeration effluent or contact stabilization effluent. As in previous runs, odors tended to increase with increasing operating temperature.

The products produced under high temperature and pressure conditions had a definite hazy appearance. With this one exception, all effluents tested under all operating conditions produced products having a crystal clear appearance.

Results of COD analyses made on these products indicated that organic contamination was significantly worse than had been experienced during previous tests using cleaner feedwaters. Since we seemed to have reached a significant break-point with regards to product water quality, the decision was made to conduct no further single stage tests with poorer quality feedwater.

# Activated Carbon Tests

In order to evaluate the effectiveness of activated carbon for removal of any odors remaining after evaporation, a bench scale granular activated carbon column was set up. This unit was 1.94" I.D. and contained a 42" deep bed of Nuchar WV-W manufactured by West Virginia Pulp and Paper Co. This grade of activated carbon has a greater proportion of small pores and is specifically recommended for removal of organics causing taste and odor problems in municipal water.

Since all products produced during the initial single stage testing had some odor, it was necessary to test examples of products produced under all conditions. Each test consisted of continuously feeding the product water to the carbon column at a rate of about 1 gpm/ft<sup>2</sup> until several column volumes had been flushed through the

column. After this flushing, samples were taken of the column feed and product for analysis. The operator would also make observations to determine whether or not any odor remained in the water from the column. In some cases the opinions of several people were secured regarding the odor of the product water.

Twenty runs were made using the activated carbon column during the initial testing phase. Six were made using products from extended aeration effluent, ten using products from contact stabilization effluent, and four using products from trickling filter effluent. In all cases the odors were either eliminated or reduced to such a level that they could be detected by only a minority of the odor test panel. The worst sample tested was a product produced under high temperature and pressure conditions using trickling filter effluent. After activated carbon treatment only one out of five members of the odor test panel detected any odor at all. COD values before and after carbon treatment for this sample were 20.7 and 3.4 mg/1.

The average COD values in and out of the carbon column for the twenty runs were 4.4 and 1.35 mg/l. Omitting the trickling filter product noted above, these averages drop to 3.6 and 1.25 mg/l.

Ammonia Ion Exchange Tests

To evaluate the feasibility of removal of ammonia from the product water by ion exchange, a bench scale ion exchange column was set up. This column was 1.94" I.D. and contained 10.25" of clinoptilolite, a natural exchange resin selective for ammonium ion. In each test, the column was operated continuously until the column had been thoroughly flushed and then samples were taken of the column feed and product. Surface loading rates of 1.2-1.4 gpm/ft<sup>2</sup> were used. A total of six

tests was made using feedwater containing from 2.5-5.0 mg/l NH $_3$ -N. Ammonia in the column effluent ranged from 0.12-0.32 mg/l NH $_3$ -N. In all cases ammonia removal exceeded 90% and averaged 94.4%.

# Aeration Tests

Probably the most economical means of removing odors from the evaporator product water would be aeration. However, the effectiveness of this method was not known. To evaluate aeration for odor removal, a 13 liter sample of product water was aerated in a 5 gallon carboy using a diffuser stone to disperse the air. The product water used was produced from extended aeration plant effluent under atmospheric pressure and initially had only a mild odor. The sample was aerated up to 1.5 ft  $^3$ /gal. This air volume exceeds by an order of magnitude the amount normally used in aeration for odor removal. Little, if any, change in odor could be detected in the water.

Because of the poor results from this test, no further evaluations of aeration for odor removal were made.

# Product Water Conductivity

Conductivity measurements were made on products produced under atmospheric and pressure conditions using extended aeration effluent and contact stabilization effluent as feedwater. It was found that conductivity correlated closely with the amount of ammonia in the product for products with less than 1.5 mg/1 NH $_3$ -N. Data for 31 product samples are shown in Figure 9. A least squares regression line for these data indicates a conductivity of 1.7 x  $10^{-6}$  mho/cm at  $7^{\circ}$ C for zero ammonia. Converting this to  $20^{\circ}$ C gives a conductivity of about 2.5 x  $10^{-6}$  mho/cm. By comparison, distilled water in equilibrium with air has a conductivity of about 1 x  $10^{-6}$  mho/cm and

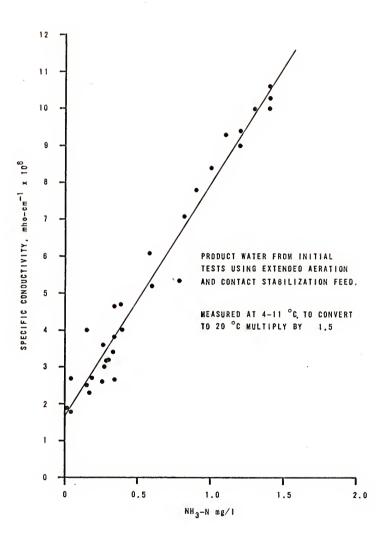


Figure 9. Conductivity vs. Ammonia Concentration

a 1.0 mg/l solution of KCl has a conductivity of about 2 x  $10^{-6}$  mho/cm.  $^{18}$ 

# Scaling During Check-Out and Initial Testing

Although there was no planned program of scale evaluation during the check-out and initial testing phase, the evaporator was mechanically cleaned several times and a few measurements made on the material removed.

During the initial check-out of the system, the evaporator was operated as a single effect unit (3rd stage only), intermittently, for a total of about 30 hours using tap water from the water treatment plant, Gainesville, Florida. Typical analysis of this water is shown in Table 15. The upper end of the evaporator was inspected by removing the vapor-liquid separator and a light tan scale was observed. The evaporator was not cleaned at this time and was operated, again on an intermittent basis, for an additional 40 hours using tap water with the pH adjusted down to 4.1-6.4 with sulfuric acid. During this period several inspections indicated that the scale was still present but did not seem to be getting any worse. At the end of this period (70 hours total operating time) the evaporator tube was mechanically cleaned with a power driven wire brush and the scale retained. A total of 16.5 grams of dry scale was removed from the evaporator. Approximate analyses on this scale indicated that it was largely calcium carbonate.

Shortly after this cleaning, the degassing equipment was installed and all feed was degassed prior to being put into the evaporator.

The evaporator was cleaned a second time after about 65 hours of operation using degassed extended aeration and contact stabilization

# TABLE 15 TYPICAL ANALYSIS OF TAP WATER, GAINESVILLE, FLORIDA

Total Hardness Calcium Hardness Magnesium Hardness Total Alkalinity Sulfate Chloride Total Dissolved Solids	95 mg/1 as CaCO <sub>3</sub> 48 mg/1 as CaCO <sub>3</sub> 47 mg/1 as CaCO <sub>3</sub> 73 mg/1 as CaCO <sub>3</sub> 32 mg/1 as SO <sub>4</sub> 20 mg/1 as C1 <sup>4</sup> 211 mg/1
•	0.

effluents. This time only 0.92 grams of scale were removed. No analyses were made on this scale.

The evaporator was cleaned a third time after the completion of the initial single stage tests. The scale removed was inadvertently discarded before it could be analyzed.

# Long Term Runs

Based on the results of the single stage tests, it appeared that no combination of effluent feedwater and evaporator operating conditions would give a product acceptable for municipal reuse without further treatment. Likewise, all product water appeared amenable to treatment by activated carbon to an acceptable quality level for reuse. Since it would be economically desirable to operate a full scale unit at as high a temperature and pressure as possible, it was decided to carry out the long term tests under the high temperature and pressure conditions used in the initial tests. All three evaporator effects were used.

The major purpose of these tests was to evaluate the scaling potential of the particular feedwater being used. Since the evaporator was operated as a three effect unit, it was possible to evaporate a greater percentage of the feedwater and thus observe the scaling that might occur in the latter stages of a full scale unit. After each extended run, the scale in each stage was mechanically removed and analyzed. In addition, the overall heat transfer coefficients for each effect were calculated as a function of time. Thus it was possible to get some indication of scaling during a run without having to shut down the equipment.

In addition to information on scaling, the long term runs gave

additional product quality data over a long period of time. Also, quality data were obtained on products from the second and third evaporator effects. Previous single effect tests had given information only on the first effect product quality.

Operating and sampling procedures during the long term runs were as follows. Fresh feed for the evaporator was prepared every 8 hours by degassing approximately 100 gallons of fresh effluent. Thus two 55 gallon drums of feed were used every 8 hours. Samples of the effluent being used, before degassing, were taken once every 8 hours and composited for 24 hours. Samples of the degassed feed and concentrated effluent were taken every 4 hours and composited for 12 hours. All the product water from each stage was collected continuously and measured and sampled each hour. The product samples were composited for 12 hours.

# First Extended Aeration Effluent Run

The first long term run was carried out using extended aeration effluent. It was planned to operate with 300°F steam and adjust the third stage pressure to evaporate about 75% of the incoming feed. The evaporator was operated for a total of 323 hours (13 days 7 hours). This excluded 9 hours down time to make minor repairs on the equipment. Other than these two brief shutdowns, one to tighten a leaking fitting and another to repair a liquid seal, the run was reasonably routine. Trouble was periodically encountered with maintaining the incoming steam to the first effect at 300°F. It was determined that there were actually two problems. First, the first effect pressure was too near the supply line steam pressure, and at times there was not enough pressure differential across the steam regulator to reliably maintain

the first effect pressure. Second, the steam trap in the condensate line from the first stage was oversized and allowed a 5-10 degree temperature drop every time it opened to discharge condensate. Consequently, on the eighth day of the run the steam temperature to the first effect was lowered to 292°F. This gave better control, but some temperature variation was still caused by the oversized trap. This problem had little effect on the validity of the scaling or product quality results, but it did make it rather difficult to determine individual heat transfer coefficients.

During the run an attempt was made to maintain a temperature drop of 32-34°F from incoming steam to third stage vapor-liquid separator temperature. At a constant feed rate of 630 ml/min this gave an average product rate of 422 ml/min or 67% of the feed rate. The product rates are shown as a function of time in Figure 10. Problems with steam control caused some variations in product rates. However, there were other variations that could not be accounted for by this factor. Overall, there was a slight increase in the total product rate over the run. This increase was due to increases in the rates from the second and third effects. These increases apparently were the result of decreased heat losses which resulted when the slightly damp insulation on these effects dried. There was a cycling pattern in the output rate that occurred within the individual effects as well as with the total. It was noted that the product rate occasionally increased markedly immediately following some type of temporary operational upset. Also, there were times that the concentrated effluent contained unusually high levels of dark-colored, suspended solids.

The analytical results for samples taken during this run are shown in Table 16.

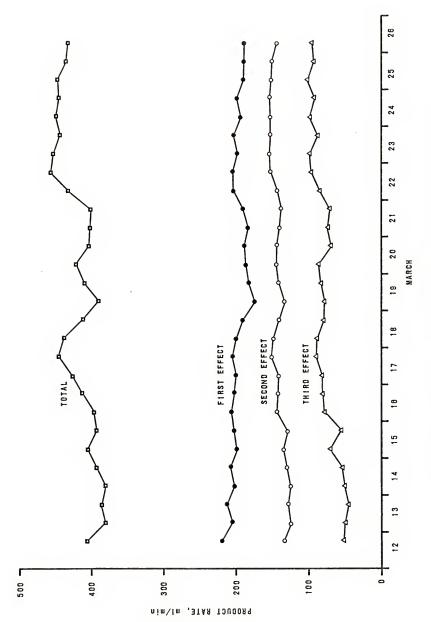


Figure 10. Product Rates, First Extended Aeration Effluent Run

TABLE 16

ANALYTICAL RESULTS - FIRST EXTENDED AERATION EFFLUENT RUN

		COD	NH3-N	NO3-N	Total Dissolved Solids <sup>a</sup>	Suspended Total Solids <sup>a</sup> Hardne	Total Hardness <sup>b</sup>	Hd	1
Feed Before Degassing	Avg. Range	46.5 35.4-63.8	0.37 0.28-0.59	17	404 398-410	20 15-24		6.6	
After Degassing	Avg. Range	39.0 28.3-49.4	0.36	18 12-24	388 354-428	16 10-20	95.3 93.6-97.8	5.5-6.2	
Concentrated Effluent	Avg. Range	105 72-211	1.4	55 42 <b>-</b> 70	1,084 938-1,222	56 34-101	317	6.2 5.8-6.6	
Products $\#1$	Avg. Range	5.9	0.12 0.05-0.19	0.14	!!	! !		!!	
#2	Avg. Range	5.0	0.13	0.10	; ;	;;			
#3	Avg. Range	4.3	0.10 0.03-0.22	0.15 0.00-0.55	; ;				
									ı

 $^3\mathrm{Spot}$  checks on less than 5 samples bAs CaCO $_3$ ; spot check on 8 feed samples and 1 concentrated effluent sample

After the completion of the run the evaporator tubes were mechanically cleaned and the scale retained. Results of the analysis of this scale are shown in Table 17.

# Trickling Filter Effluent Run

Based on the good results of the extended aeration effluent run, it was decided to make an extended run under pressure conditions using trickling filter effluent. Prior to the start of this run the oversized steam trap in the condensate line from the first stage was replaced with a smaller trap. This change made it possible to control the incoming steam temperature closer to 292°F. The operational and sampling procedures were the same as were used during the first extended aeration run.

The run lasted 285 hours (11 days 21 hours). Total down time was one hour. The feed rate was held constant at 630 ml/min. For the overall run the total product rate averaged 457 ml/min or 72.6% of the feed rate. Daily averages for the total product rate ranged from 404 to 497 ml/min or 64.2 to 79.0% of the feed rate. The overall trend in total product rate was downward. As in the previous test, cycling in the product output rate and periodic increases in suspended solids in the concentrated effluent were observed.

Analytical results for samples taken during this run are shown in Table 18.

Close control of operating temperatures made it possible to calculate accurate overall heat transfer coefficients for each effect. The 12 hour averages for these results are plotted in Figure 11. In general, the coefficients decreased with time, but tended to show a cyclic pattern.

TABLE 17 SCALE FROM FIRST EXTENDED AERATION EFFLUENT RUN

		Stage No.	
	1	2	3
Total Weight, gms	0.83	1.04	3,65
% Benzene Soluble	2.5	3.8	4.3
% Organic <sup>D</sup>	39.7	53.1	35.2
Analysis of Fired Residue			
% Soluble in 0.5N HCl	78.1	65.8	91.0 <sup>c</sup>
% Calcium as Ca <sup>d</sup> ,	14.3	11.3	12.4
% Magnesium as Mg	1.4	1.6	0.7
% Sodium as Na <sup>d</sup> % Iron as Fe <sup>d</sup>	1.0	0.7	1.3
% Iron as Fe <sup>d</sup> ,	13.5	1.8	8.0
% Sulfate as SO. ".	0.0	5.1	17.7
% Phosphate as PO <sub>4</sub> d	18.5	26.5	13.5

 $<sup>\</sup>begin{array}{l} {}^{a}_{b} \\ {}^{b} \\ {}^{b} \\ {}^{b} \\ {}^{c} \\ {$ 

TABLE 18

ANALYTICAL RESULTS - TRICKLING FILTER EFFLUENT RUN

Total Solids <sup>a</sup> pH	7.1	456 5.7 365-562 4.8-6.5	1,635 5.8 1,405-2,302 5.0-6.4		::	
T NO3-N	6.3 0.7-11	7.7 4 4.4-12 365	35 1,6 15-64 1,4	0.08 0.04-0.19	0.06	0.10
NH3-N	12.1 2.7-17	14 7.6-20	40 19-66	2.9 1.1-3.8	2.4	2.7
СОД	112 85-133	109 77-140	286 202-367	22.2 7.6-35.5	12.0 7.4-23.6	7.6
	Avg. Range	Avg. Range	Avg. Range	Avg. Range	Avg. Range	Avg.
	Feed Before Degassing	After Degassing	Concentrated Effluent	Products #1	#2	#3

<sup>a</sup>Spot checks on 5 samples each

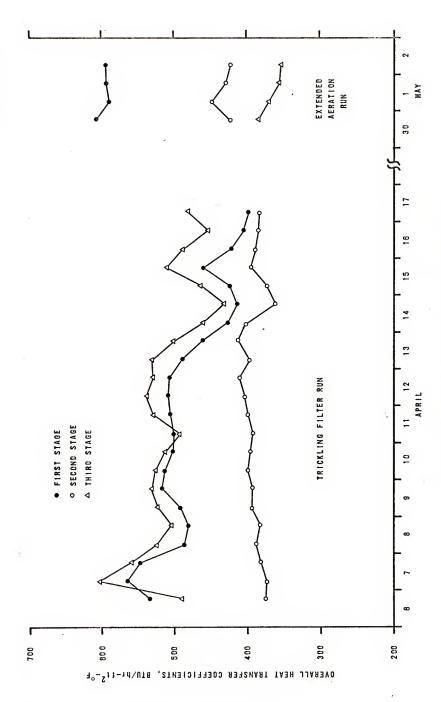


Figure 11. Overall Heat Transfer Coefficients

TABLE 19 SCALE FROM TRICKLING FILTER EFFLUENT RUN

	"Soft"	" Scale,	Stage	"Hard" Sc	ale, Stage
	1	2	3	2	3
Total Weight, gms % Organic	5.01 52.4	3.04 63.5	6.88 54.4	0.25 35.8	0.94 26.9
Analysis of Fired Residue % Soluble in 0.5N HC1 % Calcium as Cab % Magnesium as Mgb % Sodium as Nab % Iron as Fe % Sulfate as SO <sub>4</sub> % Phosphate as PO <sub>4</sub>	79.5 7.7 0.8 2.6 19.4 7.3 18.4	68.1 15.5 1.4 3.4 3.8 20.0	80.0 20.9 0.7 0.8 4.4 38.2 8.5	96.7 21.8 0.3 0.8 0.3 54.8	93.3 23.0 0.1 0.6 0.0 51.5 4.2

 $<sup>^{\</sup>rm a}{\rm Loss}$  after firing at  $600^{\rm o}{\rm C}$   $^{\rm b}{\rm Expressed}$  as percentage of soluble inorganic scale

After the completion of the run the evaporator tubes were mechanically cleaned and the scale retained. As had been experienced before, most of the scale was removed by the power-driven wire brush in less than 5 minutes. However, there was some harder grey scale in the upper portion of the evaporator tube in effects two and three. Most of this scale was removed only after considerable additional scrubbing with the wire brush. This "hard" scale was collected and analyzed separately from the previously removed "soft" scale. Results of the analysis of these scales are shown in Table 19.

To confirm the initial single stage test results with regards to odor removal, samples of each product were treated in the activated carbon column. The same column set-up used previously was used in these tests. The column loading rate for all three products was 1.0 gpm/ft. The odor was completely removed from all three products. COD results are shown in Table 20.

TABLE 20

ACTIVATED CARBON TREATMENT OF PRODUCTS FROM TRICKLING FILTER EFFLUENT RUN

	COI	)
	Before Carbon	After Carbon
	Treatment	Treatment
Product from Effect #1	20.1	4.9
Product from Effect #2	11.6	3.3
Product from Effect #3	10.6	0.1

# Second Extended Aeration Effluent Run

A reasonable explanation for the cycling in product output and heat transfer coefficients observed during the first two long term

runs seemed to be that relatively soft scale was alternately building up and then flaking off the evaporator tube walls. The periodic increases in suspended solids in the concentrated effluent was further evidence that this was occurring. To further test this theory, a second test using extended aeration effluent was made. This test was limited to 5 days. If, after 5 days, quantities of scale were found well out of proportion of what would be expected based on the previous 14 day test, this would further confirm that scale was alternately building up and flaking off.

The operational and sampling procedures used in this test were identical to those used in the first two runs. However, from the beginning of the run there was an abnormally high product rate from the third effect. The liquid seal between the second and third effects appeared to be leaking, allowing a small amount of vapor from the second vapor-liquid separator to enter the bottom of the third effect. It was felt that this would have little if any effect on the scaling rate and the decision was made not to shut down for repairs. However, the problem appeared to worsen with time, and on the third day of the run the unit was shut down for 5 hours and the liquid seal repaired. Other than the problem with this seal, the run was rather routine. The evaporator was operated for a total of 113 hours (4 days 17 hours). Total down time was 5 hours.

Analytical results for samples taken during this run are shown in Table 21.

Heat transfer coefficients for the three effects were calculated for the latter part of the run after the liquid seal was repaired. These results, calculated as 12 hour averages, are shown in Figure 11.

TABLE 21

ANALYTICAL RESULTS - SECOND EXTENDED AERATION EFFLUENT RUN

		COD	NH3-N	NO3-N	Solids	Hd
Feed Before Degassing	Avg. Range	74 38 <b>-</b> 97	0.39	14 14-15		6.8
After Degassing	Avg. Range	35 29 <b>-</b> 43	0.37 0.28-0.51	14 14-16	335 328-341	5.5-6.0
<b>Concentrated</b> Effluent	Avg. Range	136 76-203	2.3 1.6-3.7	79 62 <b>-</b> 98	1,643 1,265-2,075	5.8-5.5
$\tt Products \\ \#1$	Avg. Range	7.4	0.08	0.09		
#2	Avg. Range	4.6 1.4-8.2	0.12	0.13 0.04-0.26		
#3	Avg. Range	4.0 0.9-7.5	0.08	0.00		

<sup>a</sup>Spot check of 5 samples each

During the latter part of the run, three complete sets of samples were taken for bacteriological analyses. Results of these tests are shown in Table 22. In addition to the coliform tests, a test designed to check bacterial sterility was run on all samples. In this test the sample is incubated at 35°C for seven days in nutrient broth. This test revealed that all product samples from the first and second effects were sterile. All other samples showed some biological activity.

After the run was completed the evaporator was mechanically cleaned and the scale retained. Results of analysis of this scale are shown in Table 23.

# Three Effect Ammonia Distribution Tests

Some indication of ammonia carry over as a function of feed pH and evaporator operating conditions was gained in the single effect tests. However, additional information was needed regarding the distribution of ammonia between product and concentrated effluent in subsequent evaporator effects.

To obtain this information, a series of tests were made utilizing all three evaporator effects. Degassed contact stabilization effluent was used as the feedwater. The pH of the feed was varied from 6.0 to 8.7 and the evaporator was operated at least 4 hours under each pH condition. Product samples were taken from the last 2 hours production and composited. Feed and concentrated effluent samples were taken at the end of each run. Feed rate was held constant at 630 ml/min and the total evaporation rate varied between 447 and 545 ml/min or 71.0-86.5% of the feed rate.

The analytical results for samples taken during these runs are shown in Table 24.

TABLE 22

BACTERIOLOGICAL TESTS - SECOND EXTENDED AERATION EFFLUENT RUN

Sample	Time	Date	Total Coliform MPN/100 ml	Fecal Coliform MPN/100 m1
Extended Aeration				
Plant Effluent	2400	4/30/70	1,300,000	790,000
Feed	2400	4/30/70	< 2.0	2.0
Product No. 1	2330	4/30/70	< 2.0	<2.0
Product No. 2	2330	4/30/70	<2.0	<2.0
Product No. 3	2330	4/30/70	< 2.0	<2.0
Concentrated Effluent	2400	4/30/70	<2.0	<2.0
Extended Aeration				
Plant Effluent	0400	5/1/70	490,000	330,000
Feed	0400	5/1/70	8.0	2.0
Product No. 1	0330	5/1/70	<2.0	<2.0
Product No. 2	0330	5/1/70	< 2.0	<2.0
Product No. 3	0330	5/1/70	< 2.0	<2.0
Concentrated Effluent	0400	5/1/70	<2.0	<2.0
Extended Aeration				
Plant Effluent	0800	5/1/70	490,000	109,000
Feed	0800	5/1/70	< 2.0	<2.0
Product No. 1	0730	5/1/70	< 2.0	<2.0
Product No. 2	0730	5/1/70	< 2.0	<2.0
Product No. 3	0730	5/1/70	< 2.0	<2.0
Concentrated Effluent	0800	5/1/70	< 2.0	<2.0

TABLE 23 SCALE FROM SECOND EXTENDED AERATION EFFLUENT RUN

		Stage No.	
	11	2	3
Total Weight, gms	0.92	2.54	1.89
% Organic <sup>a</sup>	30.7	36.4	28.4
Analysis of Fired Residue			
% Soluble in 0.5N HCl	70.2	87.7	87.9
% Calcium as Ca <sup>b</sup> .	13.0	19.2	24.4
% Magnesium as Mg <sup>b</sup>	0.8	0.8	0.6
% Sodium as Nab	0.6	0.7	0.9
% Iron as Fe <sup>b</sup>	14.2	2.0	2.0
% Sulfate as SO. b	0.9	30.1	32.4
% Phosphate as PO <sub>4</sub> <sup>b</sup>	17.8	19.2	11.9

 $<sup>^{\</sup>rm a}{\rm Loss}$  after firing at  $600^{\rm o}{\rm C}$   $^{\rm b}{\rm Expressed}$  as percentage of soluble inorganic scale

TABLE 24

ANALYTICAL RESULTS - THREE EFFECT AMMONIA DISTRIBUTION TESTS

		-	2	Test No.	4	ار.	9
Feed							
After Degassing	pH NH3-N COD	6.0 8.3 35	6.7 8.0 49	7.3 8.2 53	7.6 2.8 24	8.0 9.2 26	8.7 2.3 23
Concentrated Effluent	pH NH3-N COD	5.8 35 147	18 184	 16 160	7.3 2.8 57	5.9 21 137	6.9 3.4 65
Products $\#1$	NH3-N COD	2.7	5.0	4.9 6.5	1.7	6.3	1.4
#2	NH3-N COD	3.3	2.9	2.9	1.0	3.5	1.0
#3	NH3-N COD	2.2 4.5	2.2	2.1	0.8	2.6	0.8

#### VI. DISCUSSION OF RESULTS

# Product Quality

The first objective of this work was to define the relationships between product water quality, feedwater quality, and evaporation conditions. The second objective involved defining the relationships between the above variables and post evaporation polishing. The three primary parameters used to measure product quality were ammonia content, COD, and odor. Of secondary importance in measuring product quality were such things as conductivity and nitrate content. Product quality with regard to each of these parameters is discussed separately below.

#### Ammonia Content

Ammonia is an undesirable constituent in the product water because it consumes more than six times its weight of chlorine and produces chloroamines which have much lower disinfecting efficiency than free chlorine. Previous laboratory investigations had indicated that only by going to extremely acid or basic conditions could ammonia distribution between the evaporator feedwater and the product water be controlled. In the work reported here, feedwater pH values between 5.1 and 8.7 were used in both single effect and three effect tests. The test results show that for a given pH value, the fraction of ammonia carrying over to the product increases as operating temperature and pressure increase. This is consistent with the theoretical distribution of ammonia and ammonium ion as a function of temperature.

As can be seen from the results of the single stage tests using contact stabilization effluent and trickling filter effluent, under vacuum conditions, ammonia in the product was less than 1 mg/l for feed pH values up to around 6.5. Under atmospheric conditions, feed pH had to be less than 6 to control ammonia in the product to less than 1 mg/l. It should be noted that these results are for the first effect product only. In all the single effect tests under vacuum and atmospheric conditions using contact stabilization or trickling filter effluent as feedwater, the pH of the concentrated effluent was significantly higher than the feedwater pH. This would mean that under the multieffect conditions that would be used in full scale applications, ammonia could be expected to contaminate the product water in the later stages.

Under pressure conditions, there was more than 1 mg/l ammonia in all products from contact stabilization or trickling filter feedwater for pH values down to 5.1. Tests made while operating the evaporator as a three effect unit show that this contamination persists in the second and third effects. Raising the feedwater pH as high as 8.7 did not eliminate contamination in the second and third effects.

There appear to be no easy ways of controlling ammonia contamination of the product water short of removing the ammonia from the feedwater. This could be accomplished by using a highly nitrified effluent as feedwater.

The tests reported here and the work of other investigators 51,52 show that ammonia could be removed from the product water by ion exchange using the natural resin clinoptilolite.

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# Chemical Oxygen Demand

The COD test was used as an indicator of the organic content of the product water. For the products from extended aeration effluent and contact stabilization effluent, the COD was generally less than 5 mg/l for all evaporator operating conditions. However, when trickling filter effluent was used, the product COD increased significantly as operating temperature and pressure increased. Thus it appears, that because of problems with organic carry over to the product, trickling filter effluent is a significantly poorer feedwater than either of the other two effluents tested.

The COD of products from all three effluents at all operating conditions was substantially reduced by treatment with activated carbon. However, for product water produced under pressure conditions from trickling filter effluent, the final COD after activated carbon treatment was roughly equivalent to the COD of the other products before carbon treatment.

#### 0dor

Odor in the product water will be a particularly critical factor in any full scale application of direct wastewater reuse because of the obvious psychological barriers. In these tests, no combination of wastewater feed and evaporation conditions produced completely odor free water. Product odor tended to increase in intensity and disagreeableness as evaporator temperature and pressure increased. Also, the more completely treated effluents tended to produce less odorous products.

Activated carbon was effective in removing the odor from all products. Aeration was ineffective in removing product odors.

There was some speculation regarding the effectiveness of the degassing operation in eliminating odorous compounds. Based on the relative mild odor of the degasser condensate and the odor of products from the degassed feedwater, the degassing operation seems to have little effect on product odor.

## Conductivity

Conductivity was measured on some of the products from the initial testing. As shown in Figure 9, conductivity was generally related to the ammonia content of the water. However, extrapolation to zero ammonia shows the water was of extremely high quality with regard to inorganic salts.

## Nitrate

Nitrate in the product water was used primarily as an indicator of physical carry over to the product, i.e. incomplete separation of vapor from liquid in the vapor-liquid separator. In practically all cases, nitrate test results showed that physical carry over was less than 0.5% of the incoming feedwater and in many cases much less than this.

Since it can be shown that product waters were not significantly contaminated by physical carry over, the organic contamination that did occur resulted from evaporation of volatile organics and subsequent condensation.

#### Bacteriological Tests

The bacteriological tests generally confirm that water safe for human consumption can be produced by evaporation. In all cases, product water from effects one and two was sterile. Considering this, it appears likely that the non-sterility of the product water from

effect three was due to contamination in the final condenser and receiver system. In any case, the product water would be further treated by chlorination before use.

# Evaporator Scaling

The third objective of this work was to define the relationships between feedwater quality, evaporator operating conditions, and scaling or fouling of the evaporator heat transfer surfaces.

The first indication of the potential seriousness of calcium carbonate scaling came during the initial check-out of the evaporator. Tap water that had not been degassed proved to be extremely scale forming. The 16.5 grams of scale removed from the third effect after the first 70 hours of operation was more than the total amount of scale removed from that effect during the entire remainder of the testing program. Thus it appears that degassing of evaporator feedwater to remove carbonates is essential.

The results of the three long term runs indicate that scaling from trickling filter effluent is markedly worse than scaling from extended aeration effluent. These are discussed separately below. Scaling by Extended Aeration Effluent

Two long term runs, one lasting about 13 days and the other lasting about 5 days, were made using extended aeration effluent. As explained before, the purpose of the second run was to further investigate the possibility that scale was alternately building up and flaking off in the evaporator heat transfer surfaces. The results of the second run seem to confirm this. The amount of scale in effects one and two after 5 days was more than had been removed from these two effects at the end of the 13 day run. These results, the cycling

effects observed in product output rates and heat transfer coefficients, the periodic increase in suspended solids in the concentrated effluent, and the occasional increases in product rates observed after operational upsets, all indicate that the scale was lightly adhering and would periodically flake off. Thus it appears that scaling problems with extended aeration effluent would be minor.

Analyses of the scale showed it to be about one-third to one-half organic. The inorganic portion contained significant quantities of calcium, iron, sulfate, and phosphate. It is probable that the iron came from the mixing tank and associated piping when the effluent pH was lowered for degassing. With proper materials of construction in this part of the process, iron should not be a problem. Calcium sulfate scale was formed only in the second and third effects. This reflects the increase in concentration caused by evaporation. It should be noted that, during the long term tests, conditions in the latter two effects were more severe than would occur in full scale application of this process. In a full scale plant, the evaporation of the same fraction of the incoming feed would occur over a larger number of effects and hence the temperature of the more concentrated liquid would be appreciably lower than the 260-270°F used in these tests. At the lower temperature, calcium sulfate would be less likely to form scale.

Regarding calcium sulfate scale, consideration should be given to the fact that the pH adjustment prior to degassing was made with sulfuric acid. Acid additions during the two runs ranged from 11 to 35 mg/1 SO<sub>4</sub>. If higher evaporation temperatures are to be attempted, this amount of extra sulfate could become critical.

Significant amounts of phosphate were found in the scale from all three effects. Because phosphate compounds tend to form loosely adhering sludges rather than hard scales, a high phosphate level should be desirable.

# Scaling by Trickling Filter Effluent

During the 12 day run using trickling filter effluent, the scale formed was more undesirable, both with regards to quantity and quality. Significantly more scale formed in all three effects, and the "hard" scale formed in effects two and three was very difficult to remove.

Analyses of the "soft" scale revealed that it was more than half organic, whereas the extended aeration effluent scale was generally less than half organic. The inorganic portion of the "soft" scale was very similar in composition to the extended aeration effluent scale. The "hard" scale removed from effects two and three contained a much greater inorganic fraction that appeared to be largely calcium sulfate. This finding was consistant with the physical characteristics of the material. The greater problem with calcium sulfate experienced with this feedwater may have been caused by the much larger amount of sulfuric acid that was required to lower the pH to 4.9 prior to degassing than was used in the extended aeration effluent runs. An average of 103 mg/1 SO<sub>4</sub> was added to the trickling filter effluent prior to degassing.

# Heat Transfer Coefficients

The heat transfer data in Figure 11 show that during the trickling filter effluent run, the heat transfer coefficients in effects one and three generally declined and were higher than the rather stable coefficient in effect two. During the extended aeration run, the

coefficients decreased from effect one to effect three. It is likely that during the trickling filter run the coefficients for effect three were high because of a small leak of steam from the second effect through the liquid seal. It was immediately after this run that this leak really became noticeable and was repaired. After the repair, the coefficient distribution seen in the second extended aeration run resulted.

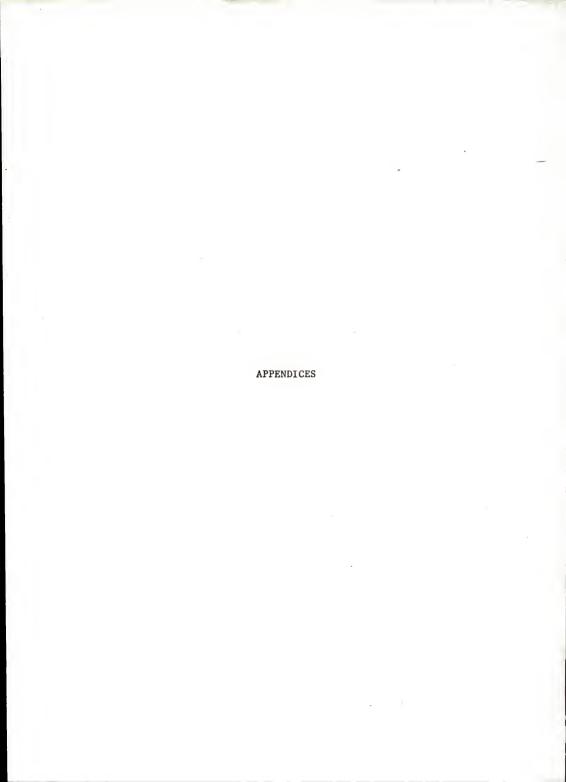
Theoretically, there should be a slight drop in heat transfer coefficient from one effect to the next due to decreasing temperature. However, the differences in coefficients seen during the extended aeration run seem to be unrealistically high. It is probable that the coefficients calculated for effects two and three are lower than the actual values. This is because the steam side temperature in these effects was measured in the vapor-liquid separator of the previous effect. Hence, any temperature drop that occurred between the vapor-liquid separator and the steam jacket of the following effect was not accounted for in the calculations. Hence, the coefficients shown in Figure 11 are of more value as relative indicators of gain or loss of heat transfer efficiency than as absolute measures of heat transfer coefficients.

#### VII. CONCLUSIONS

Based on the results of this study the following conclusions have been drawn.

- Product water produced by evaporation of extended aeration, contact stabilization, or high rate trickling filter effluent is not acceptable for reuse without further treatment because of odor and/or ammonia carry over from the feed.
- 2) Product odor tends to increase with increasing evaporator operating temperature in the range  $112-288^{\circ}F$ .
- 3) The odor of products produced from secondary effluents can be effectively removed by treatment with activated carbon. However, preliminary indications are that the odors cannot be reliably removed by aeration.
- 4) At high temperature (280-290°F) significantly more organic carry over to the product water occurs with trickling filter effluent feed than with extended aeration or contact stabilization effluent feed.
- Ammonia in the evaporator feedwater can be expected to contaminate the product water.
- 6) Removal of ammonia from the product water by ammonia selective ion exchange resins is possible.
- 7) Trickling filter effluent appears to be an unsuitable evaporator feedwater because of scaling problems.

- 8) At temperatures up to  $280^{\circ}\mathrm{F}$ , scaling problems with extended aeration effluent appear to be minor.
- 9) Evaporation of extended aeration effluent followed by activated carbon treatment gives a completely demineralized odor-free product water suitable for high quality reuse.
- 10) Because of increased efficiencies due to higher operating temperatures and negligible boiling point rise, wastewater evaporation should be more economical than sea water evaporation.



#### APPENDIX I.

#### TEST RESULTS - CAMPUS SEWAGE TREATMENT PLANT

Effluent from the High Rate Trickling Filter and the Contact
Stabilization unit of the Campus Sewage Treatment Plant was used in
this study. Shown in Tables 25 and 26 are results of tests made in
the campus plant before, during, and after the time effluent from this
plant was being used.

TABLE 25

CAMPUS SEWAGE TREATMENT PLANT TEST RESULTS,
HIGH RATE TRICKLING FILTER

		inity aCO3	Chlo as	ride Cl	ВО	D	Suspe Sol	nded ids
Date	In	Out	In	Out	In	Out	In	Out
2-18-70 2-19-70 2-25-70 2-26-70 3-4-70 3-5-70	154  160  164	82  84  76	142  146  146	140  144  126	184  212  179	47  38  36	168  166	 63  52
3-11-70 3-12-70 4-1-70 4-2-70 4-8-70 4-9-70 4-15-70 4-22-70 4-23-70 Average	148  148  142  - 150 152	74  88  72  84 80	134  132 157	192  168  144  140 151	208  162  198  190   191	51  37  36  40  41	181  136  182  125 177  162	59  18  56  44 50  49

All quantities expressed as mg/1 Initial tests were made during the period 2-27-70 to 3-3-70. The long term run was made 4-6-70 to 4-17-70.

TABLE 26 CAMPUS SEWAGE TREATMENT PLANT TEST RESULTS, CONTACT STABILIZATION PLANT

	Alkal as C	inity aCO3	Chlo as		ВО	D	Suspe Sol	
Date	In	Out	In	Out	In	Out	In	Out
2-4-70 2-5-70 2-11-70 2-12-70 2-18-70 2-19-70 2-25-70 2-26-70 3-4-70 3-5-70 3-11-70 3-12-70	120  108  136  140  128 	118  112  114  120  116  106	128  132  134  136  142  162	120  140  128  126  120  138	202  168  185  184  187  209	17.7  10.4  11.7  9.5  7.1	152  179   139  165 	15.0  15.7  14.5  8.7  11.2
Average	123	114	139	129	189	10.5	160	13.0

All quantities expressed as mg/1 The initial tests were made during the period 2-11-70 to 3-5-70.

#### APPENDIX II.

#### CALCULATION OF HEAT TRANSFER COEFFICIENTS

In calculating overall heat transfer coefficients for the evaporator the following procedures were used.

For the first effect, where the feed entered considerably below its boiling temperature, two coefficients were involved. The first coefficient was for heat transfer from condensing steam to water. The second coefficient was for heat transfer from condensing steam to boiling water and theoretically should be much larger than the first coefficient.

To solve for the two coefficients it was necessary to operate the evaporator at two slightly different steam temperatures. Assuming that the coefficients were constant over the temperature range used, the two coefficients were calculated as follows.

	Condition 1	Condition 2
Steam Temperature	T <sub>S1</sub>	T <sub>S2</sub>
Boiling Temperature	T <sub>B1</sub>	T <sub>B2</sub>
Feed Temperature	$^{\mathtt{T}}_{\mathtt{F}1}$	${\tt T_{F2}}$
Feed Rate, gpm	$\mathbf{F}_{1}$	$\mathbf{F}_{2}$
Product Rate, gpm	P <sub>1</sub>	P <sub>2</sub>

Heat transfered in the heating the feed to boiling was designated  $\boldsymbol{Q}_{\boldsymbol{h}}$ 

$$Q_{h} = (F)(8.34)(60)(T_{B}-T_{F})$$
 BTU/hr (10)

If the total area for heat transfer was A and the area used for heating the liquid to its boiling point was  $\mathbf{A}_{\mathbf{h}}$ 

$$Q_{h} = U_{h}A_{h} \overline{\Delta T_{L}}$$
 (11)

where  $U_h$  = overall heat transfer coefficient for heating

 $\overline{\Delta T_{I}}$  = log mean temperature difference

$$= \frac{(T_S - T_F) - (T_S - T_B)}{1n - \frac{(T_S - T_F)}{(T_S - T_B)}}$$
(12)

Heat transfered in boiling the liquid was designated  $\boldsymbol{Q}_{\boldsymbol{R}}$ 

$$Q_R = (P)(8.34)(60)(h)$$
 BTU/hr (13)

where h = heat of vaporization at  $T_R$ 

Then

$$Q_{B} = U_{B}(A-A_{h})(\Delta T)$$
 (14)

where  $\mathbf{U}_{\mathbf{R}}$  = overall heat transfer coefficient for boiling

$$\Delta T = T_S - T_B \tag{15}$$

Assuming that the coefficients are constant over the temperature range used for condition one and two, equations (11) and (14) can be expressed as follows

$$u_{h} = \frac{Q_{h1}}{(A_{h1})(\overline{\Delta T}_{L1})} = \frac{Q_{h2}}{(A_{h2})(\overline{\Delta T}_{L2})}$$
 (16)

$$U_{B} = \frac{Q_{B1}}{(A-A_{h1})(\Delta T_{1})} = \frac{Q_{B2}}{(A-A_{h2})(\Delta T_{2})}$$
(17)

Since for the right hand portions of equations (16) and (17) only  $A_{h1}$  and  $A_{h2}$  are unknown, these two equations can be used to solve for  $A_{h1}$  and  $A_{h2}$  and hence for  $U_h$  and  $U_B$ .

This was done for several sets of conditions, and it was found that  $\mathbf{U}_{h}$  was consistently about three times as great as  $\mathbf{U}_{R}$ .

Using this ratio of  $\mathbf{U}_h$  to  $\mathbf{U}_B$ , the coefficients can be solved for at any single condition as follows.

$$U_{h} = \frac{U_{B}}{3} = \frac{Q_{h}}{(A_{h})(\overline{\Delta}T_{L})}$$
 (18)

$$U_{B} = \frac{Q_{B}}{(A-A_{b})(\Delta T)}$$
 (19)

Substituting and simplifying

$$A_{h} = \frac{(3) (Q_{h}) (\Delta T) (A)}{(Q_{B}) (\overline{\Delta T_{L}}) + (3) (Q_{h}) (\Delta T)}$$
(20)

Substituting  $A_h$  in equation (18) or (19) gives  $U_B$ .

For the second and third effects where the liquid enters at very near its boiling point, the assumption was made that only heat transfer from condensing steam to boiling water was occurring and

$$U_{B} = \frac{Q_{B}}{(A)(\Delta T)}$$
 (21)

To save time, all calculations were made using this procedure on an IBM 360 computer.

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#### BIOGRAPHICAL SKETCH

James Haddon Sullivan, Jr., was born on April 3, 1937, in Claxton, Georgia. He graduated from Wayne County High School in Jesup, Georgia in June, 1955. The following fall he entered the Georgia, Institute of Technology in Atlanta and received a Bachelor of Chemical Engineering degree, with honors, in June, 1959.

From June, 1959, to April, 1963, he was employed as a process engineer by Union Bag-Camp Paper Corporation in Savannah, Georgia. He worked in all areas of pulp and paper making with major interest in speciality processed papers such as creped, waxed, polyethyene coated, and asphalt laminated papers. From September, 1960, to March, 1961, he was on active duty with the U. S. Army as a part of the Georgia National Guard. The remaining six years of his military obligation were fulfilled as a national guardsman.

In April, 1963, he was employed as a development engineer by the Tennessee Corporation in their Research and Development Department in College Park, Georgia. Here he worked on many research and development projects from both a technical development and economic evaluation viewpoint. In addition to his work on the development of in-house ideas, he spent considerable time in the evaluation of processes being considered for acquisition by his company.

In January, 1967, he entered the Graduate School at the University of Florida in Environmental Engineering. He received his Master of Science in Engineering degree in March, 1968. The subject of his

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This dissertation was prepared under the direction of the chairman of the candidate's supervisory committee and has been approved by all members of that committee. It was submitted to the Dean of the College of Engineering and to the Graduate Council, and was approved as partial fulfillment of the requirements for the degree of Doctor of Philosophy.

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